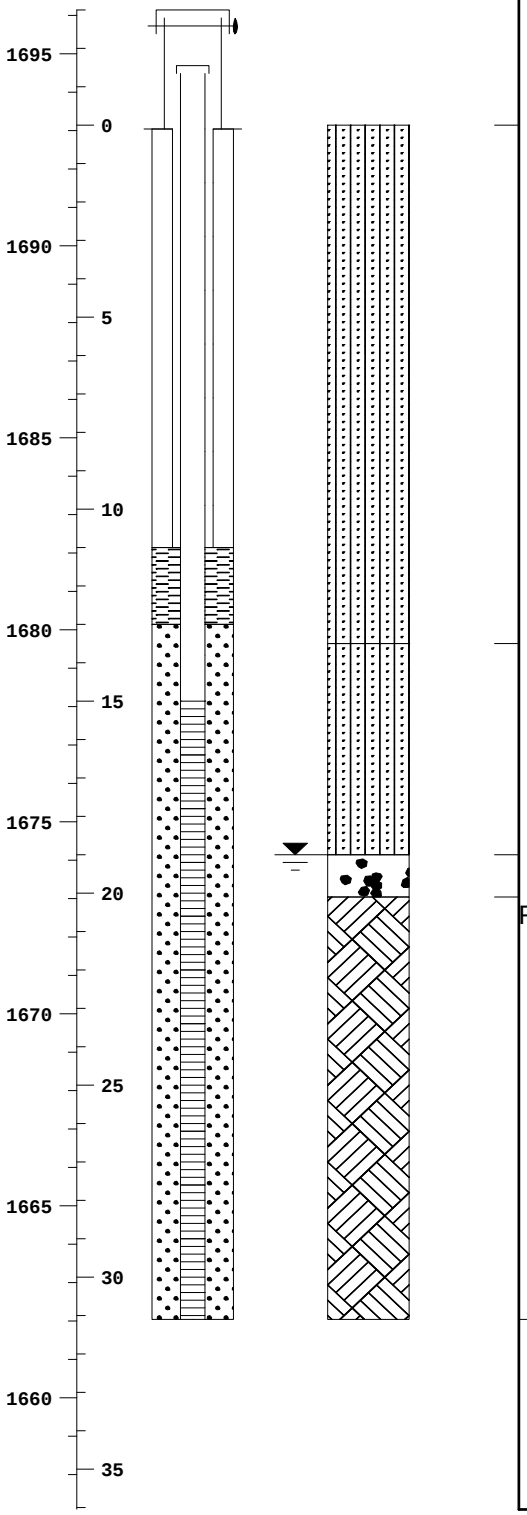
The appendices for this report are included on a CD following this page.

Appendix A-1
Boring Logs

DRILL HOLE LOG
DRILL HOLE NO.: 54MW11

PROJECT: Radford AAP SWMU 54
CLIENT/OWNER:
HOLE LOCATION: SWMU 54
DRILLER: Parrat-Wolfe
DRILL RIG: Air Rotary
DEPTH TO WATER: 19.0
HOLE DIAMETER: 2"

PROJECT NO.: 136880
DATE: 6/8/11
TOC ELEV.: 1696.2
GS ELEV.: 1693.14
LOGGED BY: J. Hillebrand
HOLE NO.: 54MW11



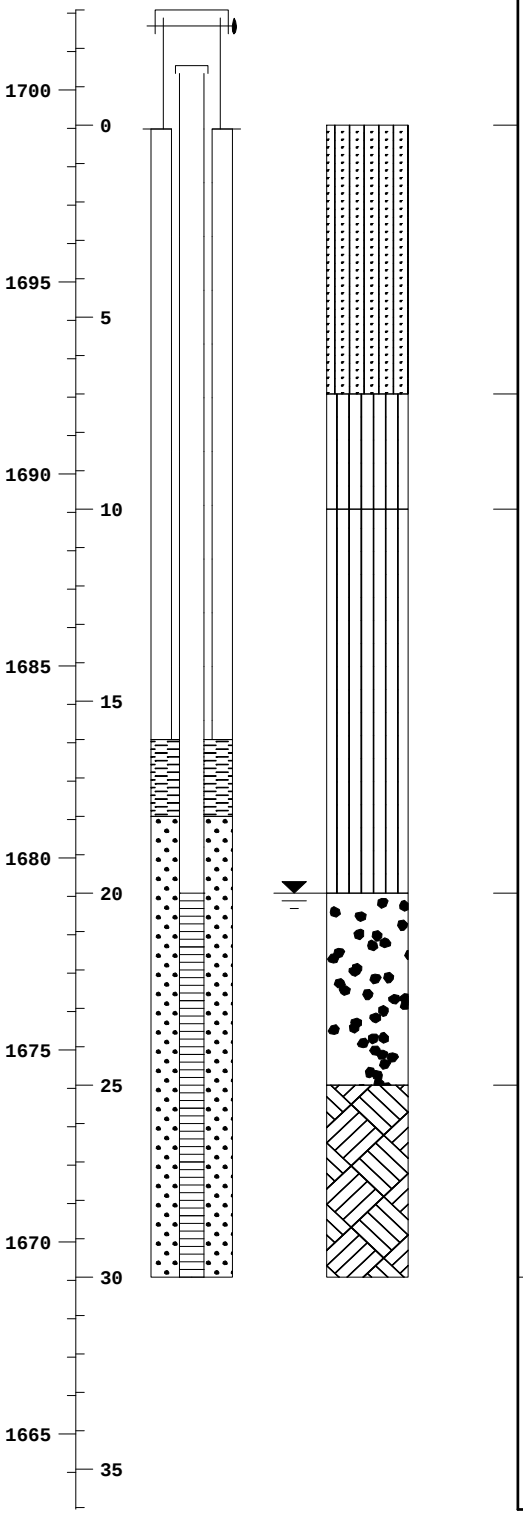
ELEVATION / DEPTH	WELL DETAILS	SOIL SYMBOLS, SAMPLER SYMBOLS AND FIELD TEST DATA	USCS	DESCRIPTION	Sample Number	Recovery	Water Content
			SM	0 Brown, moist, Silty SAND			
			SM	13.5 Brown, wet Silty SAND			
			GP	19 Gray, wet, Poorly-graded Gravel (rock fragments)			
			ROCK	20.1 Auger Refusal @ 20.1 ft bgs. Air Rotary Drilling to 30.1 ft bgs. Gray, Limestone			
				31.1			

FIGURE NO.

DRILL HOLE LOG
DRILL HOLE NO.: 54MW12

PROJECT: Radford AAP SWMU 54
CLIENT/OWNER:
HOLE LOCATION: SWMU 54
DRILLER: Parrat-Wolfe
DRILL RIG: Air Rotary
DEPTH TO WATER: 20 **HOLE DIAMETER:** 2"

PROJECT NO.: 136880
DATE: 6/9/11
TOC ELEV.: 1702.4
GS ELEV.: 1699.08
LOGGED BY: J. Hillebrand
HOLE NO.: 54MW12



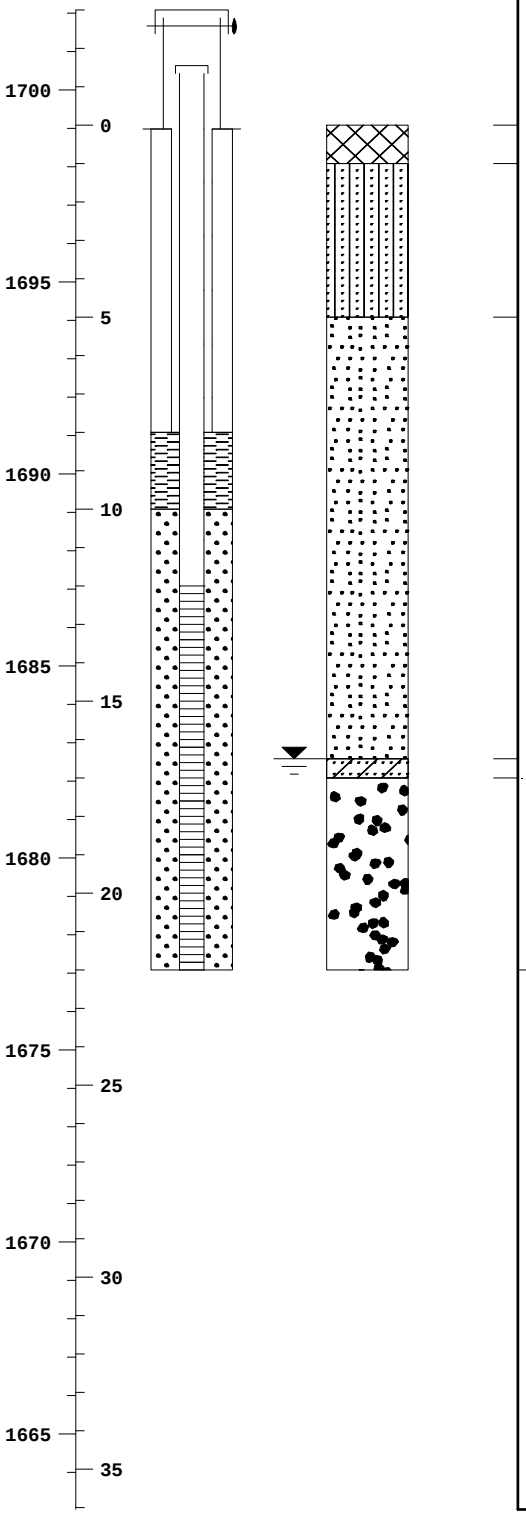
ELEVATION / DEPTH	WELL DETAILS	SOIL SYMBOLS, SAMPLER SYMBOLS AND FIELD TEST DATA	USCS	DESCRIPTION	Sample Number	Recovery	Water Content
			SM	0 Brown, moist, Silty SAND			
			ML	7 Brown, very moist, SILT with sand			
			ML	10 Brown, moist, SILT with sand			
			GP	20 Brown, wet, Poorly-graded Gravel (Rock Fragments)			
			rock	25 Auger refusal at 25 ft bgs. Air Rotary drilling to 30 ft bgs. Limestone bedrock.			
				30			

FIGURE NO.

DRILL HOLE LOG
DRILL HOLE NO.: 54MW13

PROJECT: Radford AAP SWMU 54
CLIENT/OWNER:
HOLE LOCATION: SWMU 54
DRILLER: Parrat-Wolfe
DRILL RIG: Air Rotary
DEPTH TO WATER: 16.5
HOLE DIAMETER: 2"

PROJECT NO.: 136880
DATE: 6/6/11
TOC ELEV.: 1698.9
GS ELEV.: 1699.08
LOGGED BY: J. Hillebrand
HOLE NO.: 54MW13



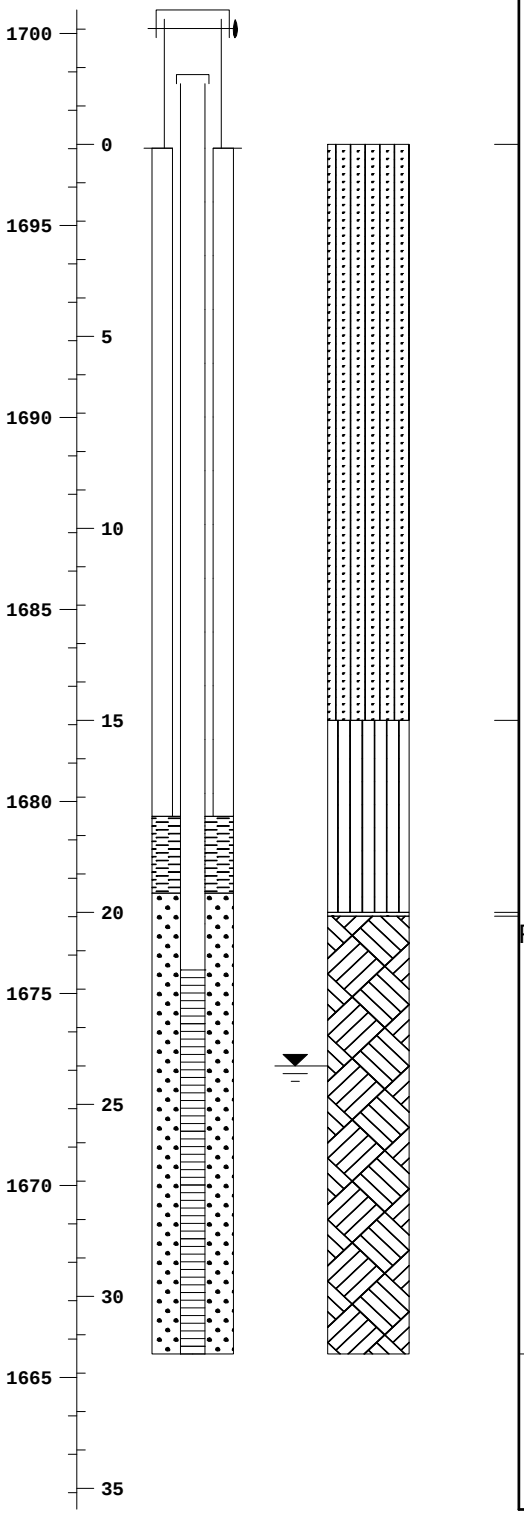
ELEVATION / DEPTH	WELL DETAILS	SOIL SYMBOLS, SAMPLER SYMBOLS AND FIELD TEST DATA	USCS	DESCRIPTION	Sample Number	Recovery	Water Content
			FILL	0 Black, dry, FILL (Road Gravel) 1			
			SM	Light brown, very moist, Silty SAND			
			SP	5 Light brown, miost, Poorly-graded SAND with silt seams; fining downwart to Silty SAND			
			SC	16.5 Gray/brown, wet Clayey SAND 17			
			GP	Brown, wet,Silty SAND Brown, wet, Poorly-graded Gravel (Rock Fragments) 22			
				Auger Refusal @ 22.0 ft bgs			

FIGURE NO.

DRILL HOLE LOG
DRILL HOLE NO.: 54MW14

PROJECT: Radford AAP SWMU 54
CLIENT/OWNER:
HOLE LOCATION: SWMU 54
DRILLER: Parrat-Wolfe
DRILL RIG: Air Rotary
DEPTH TO WATER: 24.0
HOLE DIAMETER: 2"

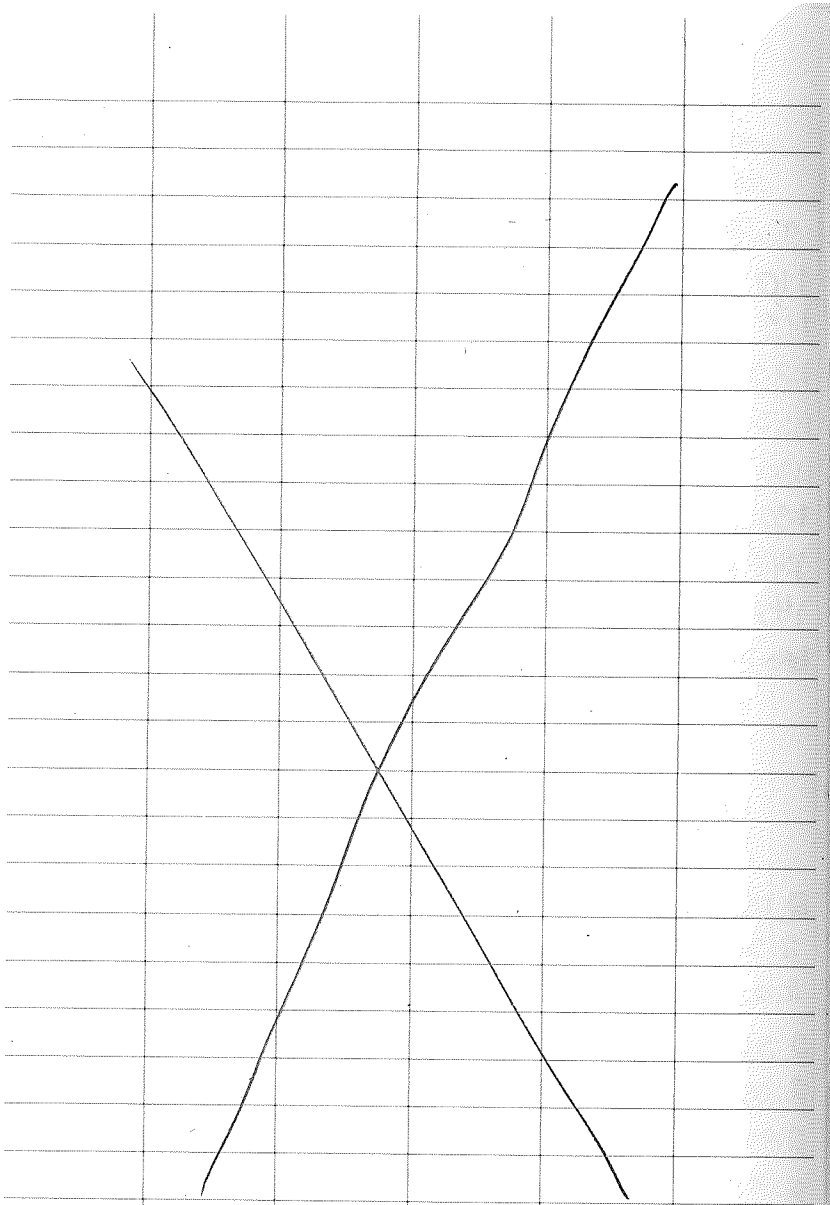
PROJECT NO.: 136880
DATE: 6/8/11
TOC ELEV.: 1700.6
GS ELEV.: 1697.11
LOGGED BY: J. Hillebrand
HOLE NO.: 54MW14



ELEVATION / DEPTH	WELL DETAILS	SOIL SYMBOLS, SAMPLER SYMBOLS AND FIELD TEST DATA	USCS	DESCRIPTION	Sample Number	Recovery	Water Content
			SM	0 Brown, moist, Silty SAND			
			ML	15 Brown, wet, Sandy SILT			
			ROCK	20 Auger Refusal @ 20.0 ft bgs. Air Rotary drilling to 31.5 ft bgs. 20.1 Grey Limestone			
				31.5 Well Set @ 31.5 ft bgs			

FIGURE NO.

Appendix A-2
Field Sampling Forms



2.19.13

S. H. Hebrard

B. Groves

Arrived onsite @ 8:00 for site safety meeting. Discussed: Cold weather, snow, slip/trip/fall, & pinch point hazards. While waiting for the site permitting meeting we calibrated the horiba according to specs. The horiba passed the calibration standards. The permitting meeting took place at 9:00 AM. Upon completion of the meeting, water levels were collected from all the well locations w/in the plant.

MW1	18.98	MW8*	13.25
MW2	22.77	MW9*	17.42
MW3	22.06	MW10*	15.32
MW4	14.24	MW11*	17.72
MW5	14.69	MW12	23.72
MW6	39.40	MW13	22.22
MW7	14.68	MW14	21.52

* collected 2.20.13

Upon completion of the GW Survey, the ~~crew~~ pump was set up on SYMW6. Well was dry in previous Sampling event.

2·19·13

MW6

well set @ 41.0' pumped @ 150 mL/m

Time	pH	cond	Turb	DO	Temp	Orp
9:45	6.67	.332	98.0	9.87	13.04	138
9:50	6.86	.320	103	9.48	13.04	121
9:55	7.06	.310	90	9.24	13.04	95
10:00	7.08	.304	54	9.24	13.07	78
10:05	7.07	.296	51	9.26	13.12	72
10:10	7.08	.293	44.2	9.28	13.14	71
10:15	7.08	.293	44.3	9.28	13.15	72

Sampled @ 10:15. Field tested for Fe + Mn

Fe = <.2 Mn = .05

Finished Sampling @ 10:35. Decomed supplies Mob to 54mw1.

Set up on 54mw1 @ 10:45. Pump set @ 45 ft. Pump flowing @ 200 mL/m.

MW1

T.me	pH	cond	Turb	DO	Temp	Orp
10:50	7.56	.447	∅	2.03	12.17	56
10:55	7.72	.465	0.0	0.61	12.05	9
11:00	7.81	.465	0.0	.73	12.12	4
11:05	7.83	.465	0.0	.71	12.03	-4
11:10	7.84	.465	0.0	.70	12.07	-5
11:15	7.85	.465	0.0	.71	12.09	-5

Sampled at 11:15. Field tested for Mn + Fe

Fe = <.2 Mn = .05

Also sampled Dup. 54TMO1

2·19·13

Sample collection finished @ 11:55. Decomed supplies. Mob To 54mw4

Set up on 54mw4 @ 12:05

pump set @ 375 ft. flow rate = 200 mL/m

MW4

Time	pH	Cond	Turb	Do	Temp	Orp
12:05	7.58	1.03	21	.88	12.15	-6
12:10	7.57	1.03	19	.74	12.18	-29
12:15	7.55	1.01	15	.56	12.20	-46
12:20	7.53	1.01	16	.51	12.14	-55
12:25	7.53	1.01	15	.56	12.09	-57
12:30	7.52	1.01	16	.53	12.06	-56
12:35	7.52	1.01	16	.53	12.09	-56

Sampled at 12:35. Field tested for Mn + Fe

Fe <0.2 Mn <05

Sample collection finished @ 12:55. Decomed supplies

Mob to 54mw7

Set up on 54mw7 @ 1:00. Pump set @ 17 ft. flow rate = 150 mL/m

MW7

Time	pH	Cond	turb	Do	Temp	Orp
1:00	7.49	.772	29	.99	11.72	-4
1:05	7.49	.731	24	1.24	11.63	-1
1:10	7.45	.608	19	2.78	11.50	8
1:15	7.42	.491	14	2.79	11.51	10
1:20	7.41	.468	17	2.81	11.49	11
1:25	7.41	.473	15	2.84	11.48	11
1:30	7.40	.474	16	2.83	11.48	12

2.19.13

Finished sampling at 1:30. Field tested for Fe & Mn

Fe <.2 Mn .10 Decomed supplies

Mob to 54MW 14

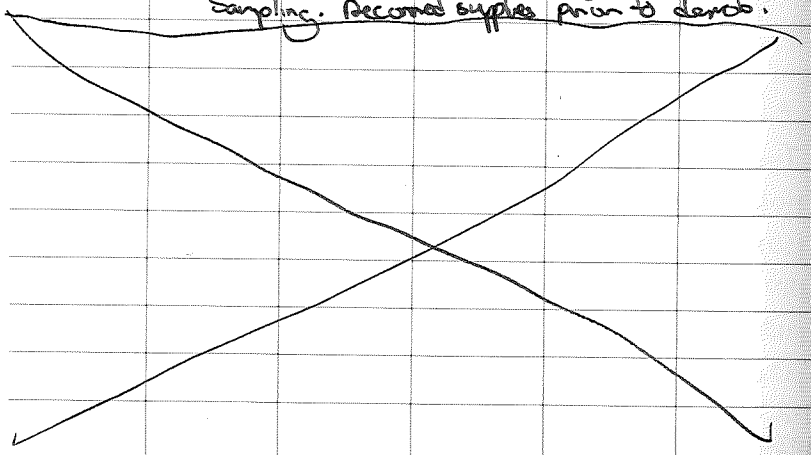
Set up on 54MW14 @ 1:40. Pump set @ 27 ft. flow rate of 150mL/min

Time	PH	Cond	Turb	DO	Temp	Orp
13:40	6.97	0.01	23.4	6.18	12.66	62
13:45	7.29	0.615	7.9	0.86	12.35	59
13:50	7.37	0.618	1.9	0.53	12.29	57
13:55	7.44	0.620	0.2	0.46	12.20	57
14:00	7.45	0.620	1.0	047	12.27	56
14:05	7.45	0.619	1.2	0.47	12.26	56

Sampled @ 14:05. Field mn & fe tested.

Fe = <.2 mn = .05

Demob for today to dispose of purge water and prepare for tomorrows sampling. Decomed supplies prior to demob.



2.20.13

J. Hilebrand

B. Graves

Arrived onsite @ 7:00. Held safety meeting at 7:15. Called gate guard at 7:20 to let us into the restricted area for well sampling. During the wait, the horiba was calibrated according to specs. Calibration passed.

Set up on 54MW9 @ 8:00. Pump set at 22'. Flow rate = 200mL/min

Time	pH	Cond	Turb	DO	Temp	Orp
8:05	6.16	.514	11	7.89	8.98	214
8:10	6.44	.495	14	8.23	9.49	202
8:15	6.66	.487	17.4	8.30	9.56	196
8:20	6.88	.485	20	8.44	9.63	192
8:25	7.03	.486	18.1	8.40	9.70	192
8:30	7.04	.487	15.2	8.31	9.73	193
8:35	7.04	.488	10.9	8.29	9.74	193
8:40	7.04	.487	10.9	8.28	9.74	194

Sampled at 8:40. Field Mn & Fe tested.

Fe = <.2 Mn=.20

collected dup sample 54TMW9.

Sampling finished at 9:15. Decomed supplies.

Mob to 54MW11.

Set up on 54MW11. @ 9:35

pump set @ 25 ft. Flow rate = 200mL/min

2.20.13

S4MW11

Time	pH	cond	Turb	DO	Temp	Orp
9:35	7.06	.681	4.3	0.73	10.06	181
9:40	7.06	.681	3.0	0.67	10.22	178
9:45	7.08	.682	3.1	0.58	10.07	174
9:50	7.09	.688	3.0	0.57	10.20	170
9:55	7.09	.686	2.1	0.58	10.12	169
10:00	7.09	.687	2.0	0.58	10.14	169

well sampled @ 10:00. Field Mn & Fe.

Mn = .25 Fe = 0.2

Sampling finished @ 10:15. Decon supplies

MOB To S4MW08.

Set up on S4MW08 @ 10:35

pump set @ 25'. flow rate = 200mL/min

Time	pH	Cond	Turb	DO	Temp	Orp
10:35	7.06	0.508	19	5.65	9.46	167
10:40	7.03	0.504	14	5.36	9.85	169
10:45	7.03	0.500	10	5.38	9.81	169
10:50	7.04	0.497	10	5.37	9.31	168
10:55	7.02	0.498	9.6	5.34	9.84	168
11:00	7.02	0.498	9.1	5.37	9.89	168
11:05	7.03	0.498	9.2	5.36	9.89	168

well sampled @ 11:05. Field Mn+Fe

Mn = .05 Fe = 0.2

* MS/MSD Collected

Sampling finished @ 11:50

Decomed supplies & MOB to S4MW10

2.20.13

Set up on S4MW10 @ 12:05

Pump set @ 23' · Flow Rate = 200mL/min

Time	pH	cond	Turb	DO	Temp	Orp
12:05	6.83	.667	54	1.63	11.10	91
12:10	6.67	.565	34	2.79	11.14	117
12:15	6.67	.553	27	2.97	11.22	122
12:20	6.68	.545	20	2.98	11.24	124
12:25	6.69	.543	15	2.97	11.15	124
12:30	6.69	.542	15	2.97	11.19	125
12:35	6.69	.542	13.2	2.97	11.22	125

well sampled @ 12:35. field test for mn & fe.

Fe = <.2 Mn = .10

called gate guard prior to sampling ^ to decon (OH) to let us out. sampling finished @ 12:50.

Drove water to treatment plant & took restroom break.

Set up on S4MW05 @ 1:10

pump set @ 19'. flow rate = 200mL/min

Time	pH	cond	Turb	DO	Temp	Orp
1:10	6.84	.414	2.3	9.53	10.65	138
1:15	6.61	.415	0.0	9.40	11.20	159
1:20	6.56	.416	0.0	9.25	11.18	166
1:25	6.55	.416	0.0	9.17	11.22	168
1:30	6.56	.417	0.0	9.21	11.18	168
1:35	6.56	.417	0.0	9.19	11.18	168

collected sample at 13:35 field tested for mn+fe

Mn .05 fe <.2

2-20-13

finished collecting 54mw05 @ 1350. Decon & mob to 54mw3.

Set up on 54mw3 @ 13:55.

pump set @ 25'. Flow rate = 200mL/min

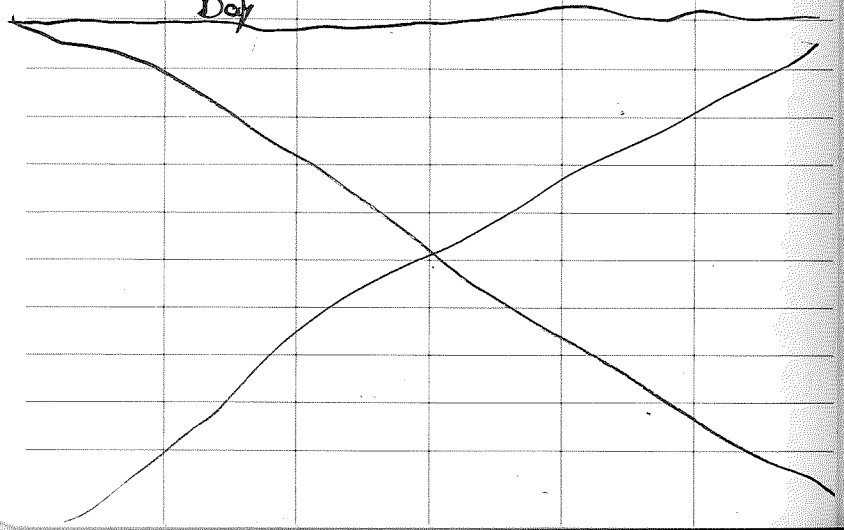
Time	pH	Cond	Turb.	Do	temp	orp
13:55	6.88	.484	29	7.51	12.02	166
14:00	6.82	.497	17.6	7.00	11.98	168
14:05	6.78	.499	13.1	6.98	11.98	170
14:10	6.79	.504	10.4	6.97	12.02	171
14:15	6.77	.504	10.0	6.97	11.99	171
14:20	6.76	.504	9.7	6.97	11.98	171
14:25	6.76	.504	9.0	6.97	11.98	171

Collected samples @ 14:25.

field mn + fe : mn=1.0 high? fe <.2

finished samples @ 14:40.

Cleaned supplies & site. Demob for Day



2-21-13

J. Nillebrand

B. Groves

Arrived onsite @ 7:10. Held safety meeting about cold, slip/trip/fall, and pinchpoints. Horiba was calibrated according to specs. Calibration passed. Set up on 54mw13 @ 7:30. Pump set @ 24 ft. flow rate of 200mL/m.

Time	pH	Cond	Turb	Do	Temp	orp
7:35	6.99	.381	7.7	8.94	7.98	151
7:40	6.98	.368	8.6	8.41	7.96	155
7:45	6.97	.370	8.7	8.41	7.97	156
7:50	6.96	.370	8.3	8.40	7.97	157
7:55	6.96	.370	7.2	8.39	7.99	157
8:00	6.96	.370	6.8	8.39	7.98	158

Sampled well @ 8:00. field mn + fe

fe <.2 mn=.20

finished sampling @ 8:15. Decomed supplies mob to 54mw2.

Set up on 54mw2 @ 8:35

pump set @ 26'. flow rate = 200 mL/m

Time	pH	Cond	Turb	Do	Temp	orp
8:35	6.90	.681	120	1.69	7.18	159
8:40	6.89	.674	97	1.53	7.70	158
8:45	6.89	.679	74	1.68	7.99	158

54MW2 cont

Time	pH	cond	turb	DO	Temp	orp
8:50	6.89	.682	51	1.44	8.19	158
8:55	6.89	.683	34	1.43	8.25	159
9:00	6.89	.682	30	1.43	8.26	157
9:05	6.89	.682	23	1.43	8.26	157
9:10	6.89	.682	21	1.43	8.26	156
9:15	6.89	.682	20	1.43	8.28	156
9:20	6.89	.682	20	1.43	8.28	156

Sampled @ 9:20. Field mn & fe.
Fe < .2 Mn < .05

Finished sampling @ 9:35. Decon &
Mob to 54MW1a.

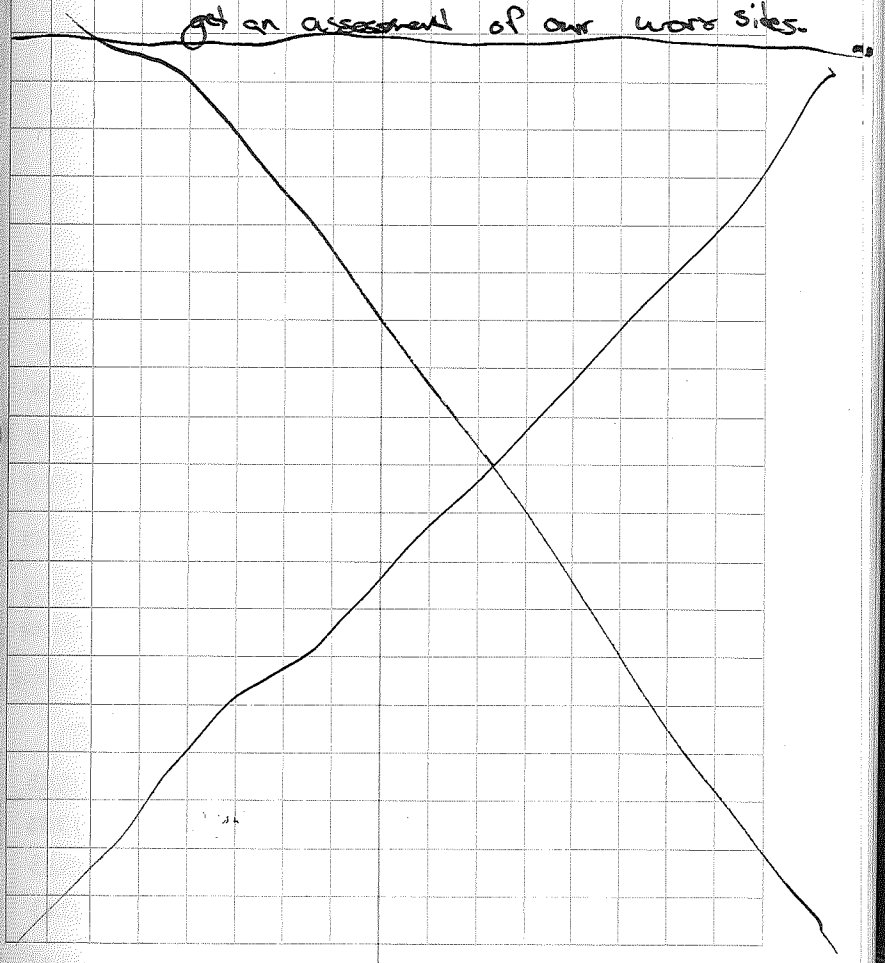
Set up on 54MW12 @ 9:55
Pump Set @ 26'. Flow rate = 200ml/m

Time	pH	cond	Turb	Do	Temp	orp
9:55	6.59	.703	108	1.58	8.26	169
10:00	6.99	.703	78	1.47	9.72	149
10:05	6.81	.583	34	1.23	10.21	152
10:10	6.50	.485	21	1.28	10.27	161
10:15	6.40	.466	20	1.29	10.25	163
10:20	6.36	.463	16	1.28	10.29	166
10:25	6.35	.463	14.3	1.28	10.29	168
10:30	6.35	.464	14.1	1.28	10.31	168
10:35	6.36	.464	14.3	1.27	10.31	168
10:40	6.36	.464	14.2	1.27	10.31	168

Sampled well @ 10:40. Field mn fe.
Mn = <.05 Fe = <.2

2.21.13

Finished sampling @ 10:55.
cleaned equipment, packed truck. Demob
from 54MW12. Water was disposed
of at the treatment facility.
Visited 54MW48/49 + Arsar to
get an assessment of our work sites.



11-6-12 J. Hilkbrand & Tucker

Arrived onsite @ 9:30. Recieved work & site entry permits.

Calibrated horiba according to SOP. Calibration was successful

JSA & Tailgate meetings were conducted

GW Levels were recorded prior to sampling

MW1	21.59	MW8	15.08
MW2	23.45	MW9	20.91
MW3	24.78	MW10	15.58
MW4	17.6	MW11	20.50
MW5	~~19.98~~ SM	MW12	26.37
→ MW6	~~Dry~~ No measurable water	MW13	~~20.21~~ 20.21 OM
MW7	18.25	MW14	23.95

* All wells have H_2O w/exception to SMMW6 **

Set up on SMMW4 pump set @ 37.5'

Pumped well @ ≈350 mL/m

Time	pH	cond	turb	DO	Temp	orp
9:45	6.59	.976	12.8	1.30	9.97	173
9:50	6.89	1.01	16.9	1.11	10.52	142
9:55	6.98	1.01	17.0	1.10	10.85	100
10:00	7.00	1.01	20.7	1.10	10.96	92
10:05	7.03	1.01	20.6	.91	11.30	77
10:10	7.03	1.01	20.7	1.01	11.26	72
10:15	7.03	1.01	18.3	1.01	11.27	73
10:20	7.03	1.01	17.0	1.00	11.26	72
10:25	7.03	1.01	17.0	1.03	11.27	72

Sampled SMMW4 @ 10:30

Field Mn = .25

Field Fe = <2

Total drawdown = ~~21.91~~ 17.91 DTW

Decomed pump & mob to SMMW7

~~Free~~ SMMW7

Pump set @ 20' pumping @ 300 mL/m

Time	pH	cond	turb	DO	Temp	orp
11:10	7.33	.835	66.2	.38	10.55	102
11:15	7.32	.832	60.9	.43	10.56	96
11:20	7.31	.831	58.3	.51	10.67	80
11:25	7.31	.832	59.7	.53	10.82	71
11:30	7.31	.830	55.3	.53	10.83	64
11:35	7.31	.830	47.6	.55	10.84	60
11:35	7.31	.829	47.0	.55	10.84	59
11:40	7.31	.829	47.0	.56	10.83	58
11:45	7.31	.828	47	.56	10.83	58
11:50	7.31	.828	46	.56	10.83	58
11:55	7.31	.826	46	.56	10.83	58
12:00	7.31	.826	46	.56	10.83	59

Sampled SMMW7 @ 10:30

Sampled SMM7 as well

Field Mn = .25

Field Fe <3

DTW = 18.42

Decomed pump & mob to SMMW14

54MW14

11.6.12

well pump set @ 28' pump @ 500 mL/m

time	pH	cond	turb	DO	Temp	orp
12:45						

Horiba failure

Horiba ceased to work. Break for Day.

Disposed of wastewater.

11.7.12 J. Hillebrand, Schriever

Held Site safety meeting @ 7:AM while waiting for replacement equipment

Equipment arrived @ 9:00.

Arrived onsite @ 10AM

Held JSA & calibrated the horiba. 4/in sec.

Set up on 54MW13

Set pump @ 21.0 ft. Pumped @ 300 mL/m

Time	pH	cond	turb	DO	Temp	orp
10:05	5.29	.605	41.9	1.02	12.21	216
10:10	5.28	.635	32.1	.98	12.23	217
10:15	5.69	.808	34.0	.97	12.24	215
10:20	5.72	.913	34.1	.96	12.27	213
10:25	5.71	.914	35.0	.96	12.25	210
10:30	5.73	.918	35.1	.97	12.25	209
10:35	5.73	.917	34.9	.97	12.25	209
10:40	5.71	.918	34.8	.95	12.26	210
10:45	5.72	.918	34.9	.96	12.25	209
10:50	5.72	.918	35	.95	12.26	209

Sample @ 10:50.

11.7.12

Fe <.2 OTW= 20.48'

Mn ~~0.3~~ 0.3 SM

Decomed equipment & MOD to 54MW9

Screen set @ 23' Pumped @ 300 mL/m

Time	pH	cond	turb	DO	Temp	orp
11:25	7.30	.782	28.0	1.25	11.23	52
11:30	7.27	.790	31.3	1.21	11.22	5
11:35	7.24	.791	30.2	1.23	11.27	-11
11:40	7.23	.792	30.6	1.12	11.29	-22
11:45	7.22	.788	29.1	1.16	11.29	-27
11:50	7.22	.787	28.3	1.14	11.30	-28
11:55	7.22	.785	28.0	1.15	11.35	-28
12:00	7.23	.784	27.8	1.14	11.34	-28
12:05	7.23	.784	27.8	1.15	11.34	-28
12:10	7.23	.781	27.7	1.14	11.35	-28

Sampled at 12:10.

Fe <.02 SM

Mn ~~0.2~~ 0.2

OTW=21.21

Decomed equipment & moved to 54MW8.

Set up on 54MW8. Set pump to 20ft

Pumped @ 450 mL/m

54MW8 11-7-12

Time	pH	cond	Turb	DO	Temp	orp
12:30	7.30	.627	5.7	2.87	11.92	99
12:35	7.29	.627	4.4	2.61	11.91	99
12:40	7.27	.627	3.7	2.73	11.89	101
12:45	7.25	.627	5.0	2.71	11.89	101
12:50	7.22	.627	3.9	2.71	11.89	101
12:55	7.22	.627	3.8	2.70	11.89	101
13:00	7.23	.627	3.9	2.71	11.90	101
13:05	7.23	.628	3.9	2.71	11.89	101

Sampled @ 13:05

Fe = <.2 MN = .05

DTW = 15.23

Decon equipment + mob to 54MW11

well set @ 25' Pumped @ ~350 mL/L

Time	pH	Cond	Turb	DO	Temp	orp
13:45	7.34	.699	1.3	1.93	12.90	119
13:50	7.32	.699	2.0	1.76	12.90	118
13:55	7.30	.699	2.1	1.63	12.91	118
14:00	7.29	.699	1.0	1.34	12.93	118
1405	7.29	.699	2.3	1.11	12.93	118
1410	7.28	.698	2.1	1.10	12.94	118
1415	7.28	.698	2.6	1.10	12.94	117
1420	7.28	.698	2.5	1.11	12.95	117
1425	7.28	.698	2.5	1.10	12.95	117
1430	7.28	.698	2.5	1.10	12.95	117

Sampled @ 14:30

11-7-12

Fe <.2

Mn .05

DTW 20.68

Decanned equipment MOB TO 54MW10

well set @ 20' Pumped @ 3cm/m

Time	pH	cond	turb	DO	Temp	orp
15:20	7.31	.771	5.3	.66	13.73	60
15:25	7.30	.773	6	.59	13.70	57
15:30	7.30	.775	5.9	.49	13.66	53
15:35	7.29	.777	5.5	.40	13.63	47
15:40	7.29	.778	5.1	.38	13.62	46
15:45	7.28	.779	5.0	.39	13.61	44
15:50	7.28	.779	4.3	.38	13.61	44
15:55	7.27	.779	4.5	.39	13.62	43
16:00	7.27	.779	4.8	.39	13.62	43
16:05	7.29	.779	4.7	.39	13.61	43
16:10	7.29	.779	4.7	.38	13.61	43

Sampled @ 16:10 also sampled 54TMW10.

Fe <.2 Mn .05

DTW = 15.81

Decon equipment + Demob. Disposed of wastewater.

11.8.12

S. Hillebrand

Etucker

Tailgate safety meeting conducted at 6AM. Discussed JSA & daily work. Arrived onsite @ 6:30. Equipment was calibrated to gear.

Set up on SMMW1. Pump set @ 45'. Pumped @ 350mL/m

Time	pH	cond	Turb	DO	Temp	orp
6:50	7.76	.428	10.0	2.93	8.84	151
6:55	7.69	.437	6.4	2.61	9.34	151
7:00	7.68	.437	5.4	2.33	9.68	151
7:05	7.66	.436	5.4	2.13	9.79	151
7:10	7.65	.440	4.4	2.00	9.81	150
7:15	7.63	.442	3.6	1.64	9.96	150
7:20	7.63	.443	3.5	1.47	10.00	149
7:25	7.63	.442	3.2	1.46	10.02	149
7:30	7.63	.443	3.0	1.45	10.03	149
7:35	7.63	.443	3.0	1.44	10.03	149
7:40	7.63	.443	3.0	1.44	10.03	148
7:45	7.63	.443	2.9	1.44	10.03	148

Sampled @ 7:45. Collected additional aliquot for MS/MSD.

Fe = <.2

Mn = .05

DTW = 21.72

11.8.12

Recovered equipment & mob to SMMW5

well set @ 20.5'. Pumped @ 350mL/m

Time	pH	cond	Turb	DO	Temp	orp
9:10	7.36	.610	8.2	1.26	10.80	165
9:15	7.18	.613	6.7	1.21	10.99	166
9:20	7.14	.615	7.8	1.16	11.15	166
9:25	7.10	.622	6.6	1.10	11.20	167
9:30	7.04	.621	6.3	1.06	11.21	167
9:35	7.01	.621	5.2	1.01	11.21	167
9:40	7.00	.621	5.5	1.01	11.21	167
9:45	6.99	.621	5.5	1.01	11.21	167
9:50	6.99	.621	5.5	1.01	11.21	167

Samples collected @ 9:50.

Fe <.2

Mn .05

Recovered equipment & Mob to SMMW14 to attempt sampling.

well set @ 28' pumped @ 400mL/m

Time	pH	cond	Turb	DO	Temp	orp
10:25	7.41	.604	104	1.52	10.40	158
10:30	7.39	.606	98.1	1.23	10.44	140
10:35	7.37	.607	76.6	1.08	10.49	126
10:40	7.32	.606	70.3	.76	10.59	113
10:45	7.31	.604	62.8	.56	10.62	103
10:50	7.30	602	50.7	.49	10.62	101
10:55	7.3	.601	43.4	.46	10.62	100

54MW14 cont. 11/8/12

Time	pH	Cond	Turb	DO	Temp	ORP
11:00	7.30	.600	43.1	.44	10.62	100
11:05	7.30	.600	43.0	.45	10.61	100

Sample collected @ 11:05

Fe <.2 MN .05

DTW = 24.21

Decomed well supplies & MOB To 54MW3

Pump set to 28' pumped @ 300ml/m

Time	pH	Cond	Turb	DO	Temp	ORP
11:40	7.10	.556	1.2	4.70	11.83	143
11:45	7.04	.565	1.2	4.61	12.10	144
11:50	7.01	.561	0.6	4.40	12.18	145
11:55	7.00	.560	0.2	4.39	12.20	145
12:00	6.97	.560	0.2	4.19	12.21	145
12:05	6.97	.561	0.2	4.08	12.21	145
12:10	6.97	.561	0.2	4.09	12.21	145
12:15	6.97	.561	0.2	4.09	12.22	145

Sample collected @ 12:15

An extra TOC sample was taken at 11:45.

FE <.2

MN .05

Decomed sample equipment & collected rinse blanks 54RB @ 12:45. MOB TO 54MW2

11-8-12

54MW2

Pump set @ 28' pumping @ 500ml/m

Time	pH	Cond	Turb	DO	Temp	ORP
1:10	7.26	.569	31.6	1.54	13.67	143
1:15	7.26	.617	15.2	1.50	13.44	142
1:20	7.21	.617	13.7	1.15	13.43	142
1:25	7.22	.618	13.7	1.01	13.43	142
1:30	7.21	.619	13.2	.94	13.42	142
1:35	7.21	.620	12.7	.89	13.42	141
1:40	7.21	.620	11.6	.86	13.43	141
1:45	7.21	.620	11.4	.86	13.43	141

Sampled @ 1:45

Fe <.2

Mn .05

Decomed equipment MOB to 54MW12

Well pump set @ 28' pumped @ 500ml/m

Time	pH	Cond	Turb	DO	Temp	ORP
2:20	7.58	.600	70.3	1.41	13.82	134
2:25	7.60	.654	37.5	1.02	13.92	134
2:30	7.59	.655	17.2	.77	13.98	134
2:35	7.60	.655	13.9	.25	13.98	135
2:40	7.60	.653	5.2	.30	13.98	135
2:45	7.60	.653	3.9	.25	13.99	135
2:50	7.60	.653	2.9	.25	13.98	135
2:55	7.60	.635	2.4	.26	13.99	135

Sampled @ 2:55. Decomed supplies. Left site to ship samples after disposing of waste water.

MW-10 MW-11 MW-12

B. Gaas
J. Mebrand

8/8/17

Held tailgate safety meeting @ 6:15. Discussed the days plans for sampling & SWMU54 & hazards associated w/ activities. Forecast is 90° w/ clouds. Arrived onsite @ 6:45. Standardized equipment according to spec. No problems. Set up on 54MW01 to begin the day.

well Set @ 6:55. 5 scfm/m

Time	pH	conductivity	Turb	Do	Temp	orp
7:00	5.99	.413	7.6	6.45	14.69	252
7:05	6.63	.399	6.3	5.99	14.25	243
7:10	6.91	.397	4.8	5.59	13.96	228
7:15	7.01	.397	5.4	5.48	13.94	224
7:20	7.09	.397	4.6	5.39	13.91	220
7:25	7.09	.396	4.1	5.36	13.90	215
7:30	7.08	.397	4.0	5.36	13.91	213
7:35	7.08	.395	4.1	5.34	13.89	212

well stabilized by 7:40. Samples collected at 7:40. 54TMW01 (dup) was also collected)

Fe <.2

Mn .05

Decon well equipment after sampling. Collected rinse blank 54RB080812 at 8:15.

MoB To 54MW06. Set up the sample Bottles & offloaded Equipment. There is not enough time to sample the well due to the meeting with the asphalt contractor. Left site @ 9:00 to get the Contractor badged in.

agree on beginning; differ on end point. End @ trailer point? mile total! Possible collapse in pipe at our original ending point. Meeting ended @ 11:10.

Set up on well 54MW06 at 11:15. 350 mL/m

Time	pH	cond.	Turb.	Do	Temp	orp	
11:15	7.60	.342	—	6.82	20.16	113	* very muddy, not much H_2O in well
11:20	7.58	.357	—	6.07	18.19	118	
11:25	7.53	.360	—	5.29	17.76	121	
11:30	7.51	.363	—	5.19	16.93	125	
11:35	7.51	.364	—	5.11	16.55	127	
11:40	7.50	.364	—	4.90	16.54	129	
11:45	7.48	.364	—	4.85	16.53	130	
11:50	7.47	.363	—	4.85	16.52	133	
11:55	7.47	.363	—	4.84	16.52	134	
12:00	7.47	.363	—	4.85	16.53	134	
12:05	7.47	.363	—	4.84	16.52	134	

Fe <.2 Mn .05

well stablized @ 12:05 w/ exception to turb.
Sampled @ 12:05. Decomed the well pump
& mob to 54mw4.
well was set @ 12:45.
54MW04 500ml/m

Time	pH	cond.	Turb	DO	Temp	orp
12:45	7.14	.851	23	.79	15.74	172
12:50	7.15	.854	20	.856	15.30	173
12:55	7.11	.877	17	.896	15.31	40
1:00	7.08	.908	15	.46	15.23	-18
1:05	7.07	.964	15	.32	15.14	-27
1:10	7.06	.955	15	.24	15.05	-33
1:15	7.06	.965	14	.23	15.06	-33
1:20	7.05	.968	13	.23	15.05	-33
1:25	7.05	.967	15	.24	15.06	-33
1:30	7.05	.968	14	.23	15.06	-33

well stablized @ 1:30. Samples were
collected @ 1:30.
Fe <.2
Mn .05
Collected MS & MSd samples 54MS8312 &
54MS8312
Decomed well pump
mob to 54MW7.

Set up on 54MW07 @ 2:25 300ml/m

Time	pH	cond	turb	DO	Temp	orp
2:25	7.17	.860	6.1	1.09	14.26	140
2:30	7.11	.848	6.2	.35	13.81	136
2:35	7.10	.843	6.7	.18	13.70	135
2:40	7.09	.839	7.0	.08	13.68	134
2:45	7.07	.833	7.6	.00	13.62	131
2:50	7.05	.823	6.8	.00	13.57	131
2:55	7.04	.820	7.6	.00	13.66	129
3:00	7.03	.819	7.4	.00	13.66	129
3:05	7.03	.817	7.7	.00	13.66	128
3:10	7.03	.817	7.4	.00	13.66	129

well stablized @ 3:10. Samples collected @
3:10.
Fe <.2 Mn=.05
well pump decomed & Mob to 54mw14.
Set up on well @ 3:40 300ml/m

Time	pH	cond	turb	DO	Temp	orp
3:40	7.09	.661	7.8	1.42	14.46	-44
3:45	7.07	.639	9.3	.55	14.17	-28
3:50	7.06	.631	9.3	.15	14.08	-19
3:55	7.06	.629	9.2	.02	14.04	-15
4:00	7.06	.627	9.5	0.00	14.05	-13
4:05	7.05	.626	9.2	.00	14.06	-13
4:10	7.05	.626	9.2	.00	14.08	-13
4:15	7.05	.625	9.2	.00	14.08	-13
4:20	7.03	.625	9.2	.00	14.08	-13

well stable @ 4:20. Sampled @ 4:20. Fe <.2 Mg <5.

Decomed well @ and mobbed offsite @ 4:45 to ~~arrived~~ ship site @ UPS

B. Groves 8.9.12

O. Hillsboro

Overcast, sunny later 90°

~~Arrived offsite~~ JSA + tailgate meeting @ 6:15

Arrived onsite @ 7:00. Calibrated equipment according to specs. No problems.

Set up on 54MW12 @ 7:15.

High initial turbidity, 3 gal/m

Time	pH	cond	turb	DO	Temp	orp
7:15	6.06	.726	—	.75	14.35	243
7:20	6.46	.707	510	.09	14.18	236
7:25	6.59	.702	151	.00	13.92	23
7:30	6.66	.686	54.5	.07	13.75	226
7:35	6.66	.676	48.2	.18	13.73	226
7:40	6.65	.667	51.5	.32	13.70	226
7:45	6.63	.667	47.7	.33	13.70	226
7:50	6.60	.665	48.1	.31	13.71	226
7:55	6.61	.665	48.2	.32	13.71	226
8:00	6.61	.665	48.0	.33	13.71	226

well stabilized @ 8:00. Samples collected @ 8:00.

Fe <.2 Mg <5

Dupe sample 54TMW12 also collected.

Decomed well supplies + mob to 54MW3

Set up on 54MW3 @ 8:30 300ml/m

time	pH	cond	turb	DO	temp	orp
8:30	6.76	.716	100	.22	13.17	209
8:35	6.81	.717	92	.00	12.97	210
8:40	6.81	.690	51	.00	12.94	209
8:45	6.79	.644	31	.00	12.94	209
8:50	6.77	.643	22	.00	12.93	209
8:55	6.77	.643	20	.00	12.93	209
9:00	6.77	.644	19	.00	12.93	209

finished stabilizing by 9:00. samples collected @ 9

Fe <.2

mg .05

Decon well pump & MOB to 54MW5 300ml/m

Time	pH	cond	turb	DO	temp	orp
9:40	6.95	.588	120	4.42	13.80	199
9:45	6.74	.584	82	4.13	13.44	203
9:50	6.71	.583	31	4.03	13.36	203
9:55	6.71	.583	25	3.95	13.41	202
10:00	6.70	.581	25	3.97	13.41	201
10:05	6.71	.581	25	3.96	13.43	200
10:10	6.71	.581	23	3.95	13.43	200
10:15	6.71	.581	23	3.96	13.42	200
10:20	6.72	.580	23	3.96	13.44	200

Sampled well @ 10:20 JH

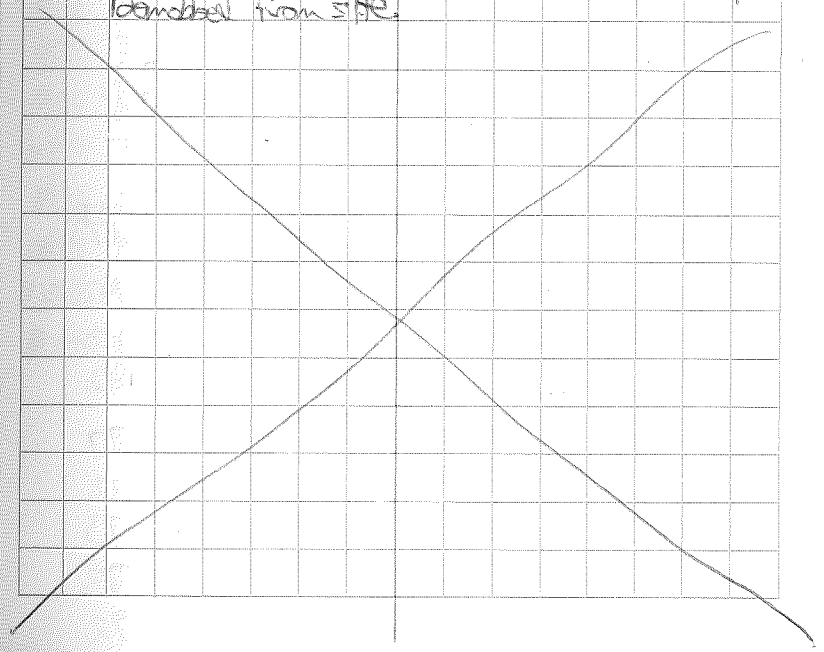
Fe<.2 mn .05

Decomed well @ 10:50. Mob to 54MW2

Mob to 54MW2. Set up on the well @ 11:35 300ml/m

Time	pH	cond	turb	DO	temp	orp
11:35	6.88	.706	180	.77	14.07	172
11:40	6.88	.727	86	.00	13.53	103
11:45	6.91	.726	71	.00	13.51	71
11:50	6.92	.725	56	.00	13.51	65
11:55	6.93	.724	43	.00	13.51	64
12:00	6.94	.723	92	.00	13.49	64
12:05	6.94	.722	44	.00	13.49	65
12:10	6.94	.722	43	.00	13.49	65

Sampled at 12:10. Decomed well pump. Demobbed from site.



5.14.13

J. Hillebrand

E. tucker

Arrived onsite @ 7:00 to wait for a vehicle permit @ the visitor center. While waiting, we held the JSA and tailgate safety meetings. ~~Set up the Stanley well pump.~~

@ Arrived onsite @ 7:45. Collected GW Levels inside fence while calibrating YSI & setting up.

54mw1	18.58	54mw9 #	15.40 #
54mw2	19.13	54mw10 #	14.20 #
54mw3	19.81	54mw11	15.60 #
54mw4	13.81'	54mw12	19.81
54mw5	12.49	54mw13	18.32
54mw6	39.62	54mw14	19.49
54mw7	13.37'		
54mw8	12.10 #		

collected 5.15.13

Set up on 54mw4. Pump set @ 39'.

Pump turned on @ 8:15. 150 mL/m

Time	Temp	cond	DO	pH	orp	Turb	▽
8:20	11.91	1.043	1.99	7.39	282.6	138	13.83
8:25	11.94	1.032	1.50	7.45	277.2	11.6	13.83
8:30	11.79	1.034	1.50	7.48	273.1	10.1	13.83
8:35	11.65	1.026	1.16	7.50	264.4	10.1	13.83
8:40	11.61	1.026	1.08	7.50	264.2	10.1	13.83
8:45	11.71	1.023	1.03	7.51	260.8	7.6	13.83

5.14.13

Time	Temp	cond	DO	pH	orp	Turb	▽
8:50	11.73	1.023	1.04	7.51	258.9	7.1	13.83
8:55	11.74	1.021	1.05	7.51	257.1	6.2	13.83

Field samples for Mn & Fe were collected. Fe = <.2 mn = .05. ≈2 gal IDW

Sample suite was collected @ 8:55 when well was stabilized. Field Dupe 54TMW01 was collected as well. Pump was removed, supplies deconned & mob to 54mw7. Pump set @ 20'. Pump turned on at 9:35. 200mL/m

54mw7

Time	Temp	Cond	DO	pH	orp	Turb	▽
9:40	12.41	.671	3.95	7.43	300.0	24.7	13.41
9:45	12.43	.671	3.97	7.42	297.4	16.9	13.43
9:50	12.56	.675	3.85	7.42	292.8	7.8	13.44
9:55	12.61	.674	3.70	7.42	290.5	5.0	13.44
10:00	12.62	.673	3.47	7.42	287.0	2.0	13.44
10:05	12.68	.674	3.25	7.41	285.8	1.7	13.44
10:10	12.71	.674	3.24	7.41	284.3	1.2	13.44
10:15	12.74	.674	3.24	7.41	282.7	0.5	13.44

well stabilized @ 10:15 Sample suite collected @ 10:15. Field Mn & Fe collected as well. Fe = <.2 MN = .05. Pulled pump. Reconned supplies & MOB to 54mw14. ≈2.7 gal IDW

5.14.13
J. Hillebrand
G Tuckey

set up on 54mw14 @ 10:40. well set @ 28'. Pump started @ 10:45. 200 ml/m

54mw14

Time	Temp	cond	DO	pH	orp	turb	▽
10:45	13.99	.613	4.69	7.32	297.3	52.4	19.52
10:50	13.34	.613	4.40	7.31	296.8	54.7	19.54
10:55	13.44	.613	4.26	7.31	296.6	0.9	19.55
11:00	13.38	.611	4.15	7.31	296.5	1.2	19.55
11:05	13.34	.610	4.15	7.31	296.4	8.7	19.55
11:10	13.41	.609	4.13	7.30	296.5	8.3	19.55
11:15	13.42	.609	4.13	7.30	296.5	7.6	19.55
11:20	13.42	.608	4.13	7.31	296.5	6.4	19.55

well stabilized @ 11:20. Field Mn & Fe samples were taken.

Fe = <.2 Mn = .05

≈2.5 gal IDW

Sample suite was collected @ 11:40. Pump was pulled & supplies decon'ed. MOB to 54mw5.

well set @ 19.5'. Pump turned on @ 11:50. 200 ml/m

Time	Temp	cond	DO	pH	orp	turb	▽
11:55	12.44	.401	4.41	6.86	246.3	9.1	12.52
12:00	12.22	.401	4.00	6.84	255.8	30.3	12.52
12:05	12.22	.399	3.53	6.83	264.8	18.5	12.53
12:10	12.16	.399	3.37	6.82	273.6	7.2	12.53
12:15	12.15	.399	3.32	6.82	275.6	5.8	12.53

5.14.13

Time	Temp	cond	DO	pH	orp	Turb	▽
12:20	12.17	.400	3.31	6.83	279.1	12.2	12.53
12:25	12.19	.399	3.29	6.83	281.4	12.3	12.54
12:30	12.20	.400	3.28	6.83	283.1	8.9	17.54

well stabilized @ 12:30. Full sample suite and field Mn + Fe were collected.

≈2.5 gal IDW

Fe = <.2 Mn = .05

~~Temp~~ Field Dupe 54TMDZ also collected at this time. Pulled pump & decon'ed equipment. MOB to 54mw1. set pump @ 45'. Pump turned on at @ 13:20. ≈200 ml/m

54mw1

Time	Temp	cond	DO	pH	orp	turb	▽
13:25	14.75	.462	1.41	7.81	202.4	6	18.61
13:30	14.80	.466	0.36	7.79	213.1	2	18.62
13:35	14.84	.465	0.23	7.79	217.2	4.3	18.62
13:40	14.52	.453	1.39	7.82	221.9	4.1	18.62
13:45	14.50	.452	1.37	7.82	222.4	4.0	18.62
13:50	14.50	.453	1.37	7.82	222.9	2.1	18.62
13:55	14.51	.452	.136	7.82	223.2	3.0	18.62

JH

well stabilized @ 13:55.

Fe = <.2 Mn = .05

Pulled pump & decon'ed equipment

Sample suite along w/ MS & MSD were collected. Drove offsite to dispose of waste water ≈ 18 gallons & to ship 3 coolers.

5.14.13
J. Hillebrand
E. Tucker

returned to site @ 4:00. MOB to 54mw3
well set @ 27'. Pump turned on @ 4:05. 200mL/s.

54mw3

Time	Temp	DO	cond	pH	orp	turb	▽
16:10	13.37	4.11	.505	7.11	247.6	31.2	19.84
16:15	13.44	3.57	.504	7.09	253.6	3.3	19.86
16:20	13.32	3.56	.503	7.07	261.7	0.4	19.86
16:25	13.26	3.57	.502	7.07	268.1	0.8	19.86
16:30	13.31	3.57	.501	7.07	271.6	1.2	19.86
16:35	13.30	3.56	.499	7.07	272.1	1.2	19.86
16:40	13.31	3.55	.499	7.06	273.4	1.3	19.87
16:45	13.31	3.55	.499	7.08	273.6	1.4	19.85

well stabilized @ 16:45
Fe: <.2 Mn: .05
±2.5 SWL JDN
Sample suite collected @ 16:45.
pump pulled & supplies decanned.
MOB To 54mw06.
well pump set @ 39.5'. pump turned on @ 17:15. 200mL/m

Time	Temp	DO	Cond	pH	orp	turb	▽
1720	13.48	.50 1.36	.503	7.35	146.2	28.1	39.65
1725	13.49	1.36	.501	7.35	144.2	17.8	39.65
1730	13.51	1.36	.498	7.34	136.8	15.2	39.65
1735	13.50	1.32	.497	7.34	133.2	13.7	39.66
1740	13.51	1.32	.497	7.34	130.1	13.1	39.66
1745	13.49	1.32	.495	7.34	129.8	11.8	39.66

5.14.13

Time	Temp	cond	DO	pH	orp	turb	▽
1750	13.49	1.30	.495	7.33	129.7	10.4	39.67
1755	13.49	1.30	.495	7.33	128.9	10.4	39.67
1800	13.49	1.29	.494	7.33	128.9	9.7	39.67
1805	13.49	1.29	.494	7.33	128.6	9.2	39.67
1810	13.48	1.29	.494	7.33	128.4	7.1	39.67

stabilized @ 1810. Sampled full suite. Fe: <.2 Mn: .05. Pulled well, decon supplies & demob for day.

5.15.13
J. Hillebrand
E Tucker

Arrived onsite @ 7:00. Called gate guard to open gate 19E. While waiting, calibrated YSI & held safety meetings. MOB To 54mw9
WL set @ 24' Pump turned on @ 7:45 200mL/m

Time	Temp	cond	DO	pH	orp	turb	▽
7:50	11.41	.521	7.17	8.00	230.3	27.5	15.44
7:55	11.39	.521	7.13	7.99	231.3	24.1	15.46
8:00	11.28	.521	7.63	7.94	236.6	10.5	15.46
8:05	11.16	.521	7.62	7.95	241.2	6.8	15.46
8:10	11.14	.521	7.61	7.95	241.8	4.0	15.46
8:15	11.12	.520	7.57	7.95	244.3	2.2	15.46
8:20	11.12	.520	7.58	7.95	244.1	4.1	15.46
8:25	11.12	.521	7.58	7.95	244.2	5.2	15.46

±2.5 SWL JDN

5.15.13
J. Hillebrand
E. Tucker

well stabilized @ 8:25. Fe= <2 Mn =.10
Sample suite collected @ 8:25. Pump pulled & supplies deconned. mob to s4mw11.

Set up on s4mw11. Pump set @ 27'.
Pump turned on @ 8:50. 200ml/m

Time	Temp	cond	DO	pH	orp	Turb	▽
8:55	12.36	.597	4.33	7.74	266.1	11.6	15.65
9:00	12.35	.596	1.53	7.61	264.2	5.6	15.65
9:05	12.41	.594	1.18	7.60	259.5	2.4	15.66
9:10	12.43	.593	1.16	7.59	257.9	1.4	15.66
9:15	12.42	.593	1.18	7.59	256.9	0.0	15.66
9:20	12.42	.593	1.18	7.60	256.4	0.2	15.66
9:25	12.42	.592	1.18	7.60	256.1	0.2	15.66
9:30	12.42	.592	1.17	7.59	255.8	0.3	15.67

well stabilized @ 9:30 final suite collected
Mn .05 Fe <.2

well pump pulled & supplies deconned.
MOB TO s4mw8.
Set up on s4mw8. well set @ 26'.
pump turned on @ 10:00. 200ml/m

Time	Temp	cond	DO	pH	orp	Turb	▽
10:05	13.37	.606	0.48	7.47	261.3	0.3	12.18
10:10	13.21	.606	0.36	7.47	258.2	0.7	12.19
10:15	13.29	.603	0.39	7.48	256.3	10.4	12.19
10:20	13.33	.596	0.40	7.48	254.9	10.5	12.19
10:25	13.37	.596	0.40	7.48	254.6	1.3	12.19

5.15.13

Time	Temp	cond	DO	pH	orp	turb	▽
10:30	13.23	.596	.41	7.47	253.7	.7	12.19
10:35	13.32	.596	.40	7.47	254.2	3.2	12.13
10:40	13.32	.596	.40	7.46	254.6	2.4	12.19
10:45	13.32	.596	.40	7.47	255.1	1.6	12.19

well stabilized @ 10:45. Fe= .2 Mn=.05
Sample suite collected @ 10:45.
Pump pulled & Supplies deconned.
MOB to s4mw10. Pump set at 23'.
Pump turned on @ 11:20. 250ml/m

Time	Temp	cond	DO	pH	orp	Turb	▽
11:25	12.47	.012	5.42	8.11	271.3	2.5	14.24
11:30	12.83	.544	2.09	6.98	277.1	2.9	14.24
11:35	12.87	.544	2.09	6.98	278.3	1.6	14.24
11:40	12.90	.543	2.41	6.97	281.0	1.0	14.24
11:45	12.90	.542	2.48	6.96	283.2	1.0	14.24
11:50	12.89	.539	2.44	6.95	284.5	4.3	14.24
11:55	12.89	.537	2.44	6.94	287.1	5.7	14.24
12:00	12.89	.537	2.43	6.94	287.3	6.2	14.24
12:05	12.89	.537	2.43	6.94	287.8	1.4	14.24

well stabilized @ 12:05. Fe= <.2 Mn=.05
Sample suite collected @ 12:05. Pump was pulled & supplies deconned. Rinse blank RB05 15 13 was collected @ 12:35.
While the rinse blank was collected, I set up on rover sample PW-9.

5.15.13
J. Hilkbrandt
E. Tucker

PW-9

Surface water

Time	temp	cond	DO	pH	Turb	orp
~~12:40~~ 12:40	19.83	.155	6.98	7.75	80.3	214.4

Pore water YSI emptied & probe inserted 8"

~~12:43~~	18.42	.346	1.45	~~7.35~~ 6	38.6	254.2

Pore water Fe <.2 Mn <.05

Time	~~Temp~~	cond	DO	pH	Turb	orp
12:45						

Samples collected @ 12:45

Bac Probe deconned. Mobs to PW8

Surface

Time	temp	cond	DO	pH	Turb	orp
1:~~15~~	16.22	.119	5.95	7.42	49.2	263.9
Pore	19.23	.343	2.67	6.88	347.4	272.4

Time	temp	cond	DO	pH	Turb	orp
1:20						

Fe <.2 Mn <.05

Sample collected @ 1320.

Decon probe mob to PW5

Surface

Time	Temp	cond	DO	pH	Turb	orp
1315	18.75	.162	2.69	7.43	31.3	236.8

Pore

Time	temp	cond	DO	pH	Turb	orp
1350	19.74	0.687	0.4	7.08	24.2	266.4

Fe <.2 Sample @ 13~~30~~. Decon & mob

Mn <.05

5.15.13

PW-3

Surface

Time	Temp	cond	DO	pH	turb	orp
14:30	18.65	.164	2.53	~~7.89~~	37.2	188.7

Pore

Time	Temp	cond	DO	pH	Turb	orp
14:35	17.81	.233	1.30	7.25	442.6	202.2

Fe <.2 sample @ 14:35. Decon & mob to PW1

Mn<.05

PW-1

Surface

Time	Temp	con	DO	pH	Turb	orp
15:25	18.60	.064 ~~12.7~~	2.71	7.55	138.6	212.2

Pore

Time	Temp	cond	DO	pH	Turb	Orp
15:30	19.60	.160	0.74	7.23	111.1	214.4

Fe<.2 Sampled @ 15:30. called gate guard to

Mn<.05 tel us in the main plant area. Disposed of water all treatment plant. Demob for day.

END

5.16.13

Thacker
J. Hillebrand

Arrived onsite @ 6:15. OSA & Safety meeting were conducted @ 6:15. MOB to 54mw2. Standardized Headspace
well pump set @ 26'. Pump turned on at 6:25.
250 mL/m 54mw2

Time	temp	cond	DO	pH	orp	turb	▽
6:30	12.48	.484	1.26	7.14	201.4	147.6	19.17
6:35	12.05	.307	2.18	6.54	227.6	25.0	19.19
6:40	12.12	.303	2.52	6.50	240.9	0.8	19.21
6:45	12.21	.312	2.74	6.51	251.5	1.7	19.22
6:50	12.22	.314	2.73	6.51	253.0	1.1	19.22
6:55	12.21	.314	2.74	6.52	253.6	1.7	19.23
7:00	12.21	.315	2.74	6.52	253.2	1.0	19.23
7:05	12.22	.315	2.74	6.52	254.0	0.5	19.23
7:10	12.22	.315	2.74	6.52	254.3	0.2	19.23

Samples collected @ 7:10 when the well had stabilized. Fe = <.2 Mn = .05 Pulled the well pump & Decomed supplies. MOB TO 54mw12.

Setup on 54mw12. well set @ 26'. Pump turned on @ 7:45. 250 mL/m

5.16.13

54mw12

Time	temp	cond	DO	pH	orp	turb	▽
7:50	13.27	.674	2.86	6.91	293.2	178.8	19.85
7:55	13.30	.670	2.59	6.80	294.3	95.7	19.87
8:00	13.27	.666	2.58	6.78	293.2	54.3	19.87
8:05	13.28	.666	2.58	6.78	292.7	17.0	19.88
8:10	13.28	.665	2.57	6.78	292.4	5.3	19.88
8:15	13.28	.665	2.57	6.78	292.5	6.2	19.89
8:20	13.28	.665	2.57	6.78	292.4	3.8	19.89

well stabilized @ 8:20. Samples collected @ 8:20. Fe=<.2 Mn = <.05
well pump pulled & supplies decomed. MOB to 54mw13.

Setup on 54mw13. well set @ 24'. pump turned on @ 8:55. 200ml/m

54mw13

Time	temp	cond	DO	pH	orp	turb	▽
9:00	13.22	.430	5.05	7.34	265.9	49.2	18.42
9:05	12.91	.427	3.46	6.92	273.8	19.9	18.44
9:10	12.84	.435	1.91	6.93	278.9	2.9	18.45
9:15	12.82	.440	1.42	6.95	280.7	2.1	18.45
9:20	12.84	.441	1.41	6.96	282.6	1.6	18.45
9:25	12.84	.441	1.41	6.97	283.1	1.1	18.45
9:30	12.84	.441	1.40	6.97	283.4	1.1	18.45
9:35	12.84	.440	1.40	6.97	284.0	0.2	18.45

5.16.13
S Hillebrand
G Tucker

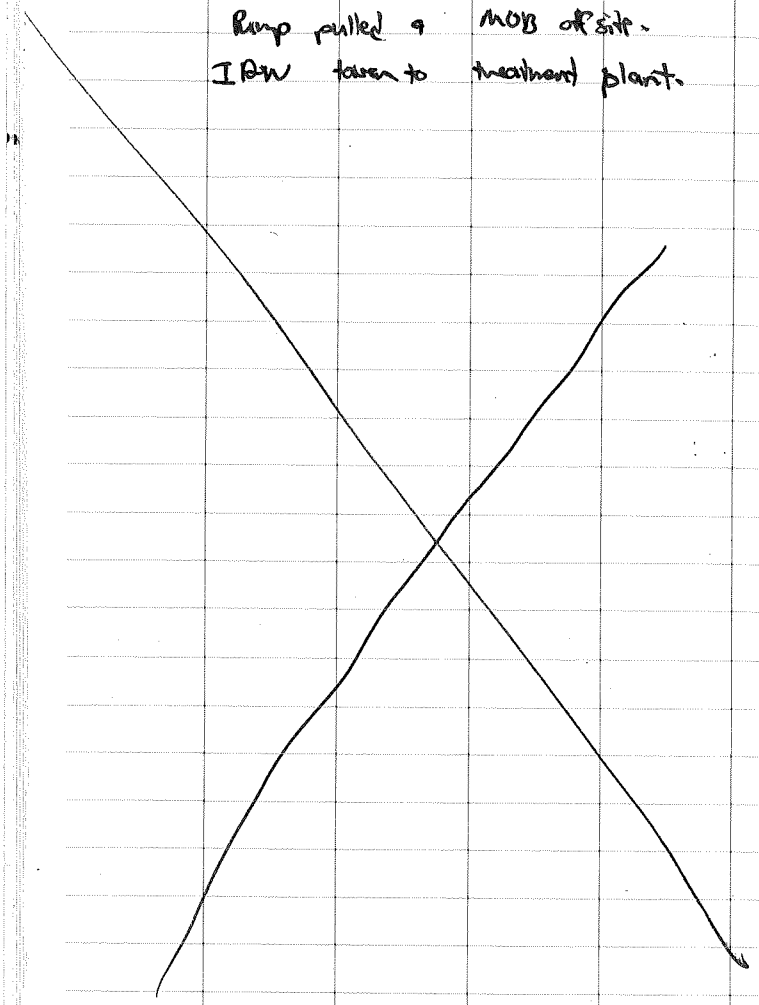
Well 34mw13 stabilized @ 9:35.

Samples collected.

$\leq 4 < .2$ $m = .15$

Pump pulled & MOB off site.

IDW taken to treatment plant.



Appendix B-1
Chains of Custody

L13050733 / Revision: 0 / 463 total pages

Page 451

Generated: 05/31/2013 15:42

COC No. A 35430

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: CBRI

Project Contact: Eric Mathews | Contact Phone #: 410 273 7237

Turn Around Requirements: | Location: SWMU 54

Project ID: Radford SWMU 54 MNA

Sampler (print): Emily Tucker / Jeff Hillebrand | Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Perchlorate	Explosives	Anions	TIC TOC H_2SO_4 preserved	TIC-O TOC-O	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54MW4		/	5.14.13	8:55	GW	6		1	2	1	1	1		
54TM01		/	5.14.13	8:55	GW	6		1	2	1	1	1		Cooler 1
54MW07		/	5.14.13	10:15	GW	6		1	2	1	1	1		
54MW14		/	5.14.13	11:20	GW	6		1	2	1	1	1		
54MW5		/	5.14.13	12:30	GW	6		1	2	1	1	1		cooler 2
54TM02		/	5.14.13	12:30	GW	6		1	3	1	1	1		
54MW1		/	5.14.13	13:55	GW	6		1	2	1	1	1		
MSD54MW1		/	5.14.13	13:55	GW	6		1	2	1	1	1		MS cooler 3
MS 54MW1		/	5-14-13	13:55	GW	6		1	2	1	1	1		MSD

Relinquished by: (Signature)	Date	Time
	5.14.13	3:30
Relinquished by: (Signature)	Date	Time

Microbac OVD
Received: 05/15/2013 09:55
By: ROSEMARY SCOTT
221000035925

Date	Time	Received by: (Signature)

Remarks:

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

L13050819 / Revision: 0 / 674 total pages

COC No. A 35429

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: CB&I (Shaw

Project Contact: Eric Malarek | Contact Phone #: 410-273-7233

Turn Around Requirements: Standard | Location: Radford AAP

Project ID: SWMU 54 MNA

Sampler (print): Jeff Hilebrand | Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explosives	Anions	Perchlorate	Tic - TOC (preserved)	TIC-D, TOC-D	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54mw3		/	5-14-13	16:45	GW	6		2	1	1	1	1		cooler 1
54mw6		/	5-14-13	18:10	GW	6		2	1	1	1	1		
54mw9		/	5-15-13	8:25	GW	6		2	1	1	1	1		
54mw11		/	5-15-13	9:35	GW	6		2	1	1	1	1		cooler 2
54MW8		/	5-15-13	10:45	GW	6		2	1	1	1	1		cooler 2
54MW10		/	5-15-13	12:05	GW	6		2	2	1	1	1		cooler 2
~~RB05153~~		/	~~5-15-13~~	~~12:30~~	GW	6		2	1	1	1	1		~~cooler 2~~
PW9		/	5-15-13	12:45	GW	6		2	1	1	1	1		
PW8		/	5-15-13	1320	GW	6		2	1	1	1	1		cooler 3
PW5		/	5-15-13	1320 1350	GW	6		2	1	1	1	1		
PW3		/	5-15-13	14:35	GW	6		2	1	1	1	1		cooler 4
PW1		/	5-15-13	15:30	GW	6		2	1	1	1	1		

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Date	Time
	5-15-13		Microbac OVD Received: 05/16/2013 14:48 By: ROSEMARY SCOTT 221000036049		
Relinquished by: (Signature)	Date	Time	Remarks:		

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

Generated: 05/31/2013 18:57

L13050918 / Revision: 0 / 476 total pages

COC No. A 35429

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: CB&I (Shaw)

Project Contact: Eric Malarek

Contact Phone #: 410-273-7233

Turn Around Requirements: Standard

Location: Radford AAP

Project ID: SWMU 54 MNA

Sampler (print): J. Hillebrand / E. Tucker

Signature:

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explosives	Anions	Perchlorate	TIC, TOC	TIC-D	TOTAL # (LAB USE)
54MW 2		/	5.16.13	7:10	GW	6		2	1	1	1	1	
54 MW 12		/	5.16.13	8:20	GW	6		2	1	1	1	1	
54MW 13		/	5.16.13	9:35	GW	6		2	1	1	1	1	

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other _____

ADDITIONAL REQUIREMENTS

Microbac OVD
Received: 05/17/2013 14:04
By: BOB BUCHANAN
221000036143

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Date	Time
	5.16.13				
Relinquished by: (Signature)	Date	Time	Remarks:		

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

Generated: 06/03/2013 15:08

L13020607 / Revision: 0 / 464 total pages

Page 456

Generated: 02/27/2013 13:57

COC No. A 37147

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: CBE (Shaw E&I)

Project Contact: Eric Mabarek

Contact Phone #:

Turn Around Requirements: Normal

Location: Radford AAP

Project ID: SWMU 54 MNA

Sampler (print): Bill Groves

Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explosives	Perchlorate	TIC-D, TOC-D	TIC, TOC	Anions	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
~~54MW6~~ 54MW6	X		2.19.13	10:15	GW	6		2	1	1	1	1		
~~[redacted]~~ 54MW1	X		2.19.13	11:15	GW	6		2	1	1	1	1		
54TM1	X		2.19.13	11:15	GW	6		2	1	1	1	1		
54MW4	X		2.19.13	12:35	GW	6		2	1	1	1	1		
54MW7	X		2.19.13	13:30	GW	6		2	1	1	1	1		
54MW14	X		2.19.13	14:05	GW	6		2	1	1	1	1		

Microbac OVD
Received: 02/20/2013 09:53
By: ROSEMARY SCOTT
221000032643

Relinquished by: (Signature)	Date	Time	Rec (Sig	Date	Time	Received by: (Signature)
	2.19.13					
Relinquished by: (Signature)	Date	Time	Rec (Sig	Remarks:		

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

2 coolers

Page 1 of 1

L13020654 / Revision: 0 / 543 total pages

COC No. A 37397

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: CB&I

Project Contact: Eric Malerak | Contact Phone #:

Turn Around Requirements: Normal | Location: RFAAP

Project ID: Swmu 54 MNA

Sampler (print): Bill Groves | Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other _____

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explosives	Perchlorate	TIC-D, TOC-D	TIC, TOC	Anions	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54MW9	X		2.20.13	9:40	GW	6		2	1	1	1	1		
54TMW9	X		2.20.13	8:40	GW	6		2	1	1	1	1		
54 MW11	X		2.20.13	10:00	GW	6		2	1	1	1	1		
54 MW 8	X		2.20.13	11:05	GW	18		6	3	3	3	3		MS/MSD → 2x volume
54 MW10	X		2.20.13	12:35	GW	6		2	1	1	1	1		
54 MW 5	X		2.20.13	1335	GW	6		2	1	1	1	1		
54 MW 3	X		2.20.13	14:25	GW	6		2	1	1	1	1		

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Date	Time
[signature]	2.20.13		Microbac OVD Received: 02/21/2013 09:42 By: ROSEMARY SCOTT 221000032679		
Relinquished by: (Signature)	Date	Time	Remarks:		

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

Generated: 03/05/2013 13:06

COC No. A 37398

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: CBEI

Project Contact: Eric Maleski

Contact Phone #:

Turn Around Requirements: Normal

Location: RFAAP

Project ID: SWMU 54 MNA

Sampler (print): B. Groves

Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other _____

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explosives	Anions	Perchlorate	TIC-D, TOC-D	Fe++, TOC	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54MW13	X		2.21.13	8:00	GW	6		2	1	1	1	1		
54MW2	X		2.21.13	9:20	GW	6		2	1	1	1	1		
54MW12	X		2.21.13	10:40	GW	6		2	1	1	1	1		
54RB22113	X		2.21.13	12:00	GW	6		2	1	1	1	1		

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Date	Time
	2.21.13		Microbac OVD Received: 02/22/2013 10:12 By: ROSEMARY SCOTT 221000032722		
Relinquished by: (Signature)	Date	Time	Remarks:		

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

L12110202 / Revision: 0 / 365 total pages

COC No. A 33671

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: Shaw E+I

Project Contact: Eric Malavet | Contact Phone #:

Turn Around Requirements: Standard | Location: SWMU 54

Project ID: SWMU 54 MNA

Sampler (print): O. Nikkebaard / E. Tucker | Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explosives	Anions	Perchlorate	TIC TOC	TIC-0	TIC-0	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54MW4	/		11.6.12	10:30	GW	6		2	1	1	1	1			
54MW7	/		11.6.12	12:00	GW	6		2	1	1	1	1			
54TM7	/		11.6.12	12:00	GW	6		2	1	1	1	1			

Relinquished by: (Signature)	Date	Time	R (S	Date	Time	Received by: (Signature)
	11.6.12	4:00				
Relinquished by: (Signature)	Date	Time	R (S			

Microbac OVD
Received: 11/08/2012 09:54
By: BRENDA GREENWALT 221000030049
Brenda Greenwalt

Remarks:

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page 1 of 1

Generated: 11/21/2012 09:10

COC No. A 33673

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: Shaw E&I

Project Contact: Eric Malarek | Contact Phone #:

Turn Around Requirements: Standard | Location: SWMU 54

Project ID: Radford SWMU 54 MNA

Sampler (print): JH/ET | Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explosives	Anions	Perchlorate	TIC TBC	TIC-10 TOC-10	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54MW 13	✓		11-7-12	10:00	GW	6		2	1	1	1	1		
54MW 9	✓		11-7-12	12:10	GW	6		2	1	1	1	1		
54MW 8	✓		11-7-12	13:05	GW	6		2	1	1	1	1		
54MW 11	✓		11-7-12	14:30	GW	6		2	1	1	1	1		
54MW 10	✓		11-7-12	16:10	GW	6		2	1	1	1	1		
54TM 10	✓		11-7-12	16:10	GW	6		2	1	1	1	1		

Microbac OVD
Received: 11/08/2012 09:54
By: BRENDA GREENWALT 221000030051

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Date	Time
	11-7-12	5:00	Brenda Greenwalt		
Relinquished by: (Signature)	Date	Time			

Remarks:

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

L12110249 / Revision: 0 / 521 total pages

COC No. A 33672

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: Shaw E&I

Project Contact: Eric Meluntk | Contact Phone #:

Turn Around Requirements: Standard | Location: SWMU 54

Project ID: Radford SWMU 54 MNA

Sampler (print): JN + ET | Signature:

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explosives	Anions	Perchlorate	TIC TDG	TIC-D, TOC-D	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54MW1	/		11-8-12	7:45	GW	18		4	3	3	3	3		MS/MSD
54MW5	/		11-8-12	9:50	GW	6		2	1	1	1	1		
54MW14	/		11-8-12	11:05	GW	6		2	1	1	1	1		
54MW3	/		11-8-12		GW	6		2	1	1	1	1		
Extra TOC	/		11-8-12	11:45	DUP	1					1			
~~54MW~~ 54RB	/		11-8-12	12:45	GW	6		2	1	1	1	1		
54MW2 [illegible]	/		11-8-12	1:45	GW	6		2	1	1	1	1		
54MW12	/		11-8-12	2:55	GW	6		2	1	1	1	1		

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by:	Date	Time	Received by: (Signature)
	11.8.12						
Relinquished by: (Signature)	Date	Time					

Remarks:

Microbac OVD
Received: 11/09/2012 10:32
By: ROSEMARY SCOTT
221000030109

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

Generated: 11/21/2012 13:56

L12080249 / Revision: 0 / 470 total pages

COC No. A 31145

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: Shaw E&I

Project Contact: 54MW 54MWA | Contact Phone #: Eric 410-270-7233

Turn Around Requirements: Standard | Location: Bel Camp Md.

Project ID: Radfed

Sampler (print): Bill Groves | Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explo E330	Alkalinity (4 Lbs)	Cyanide	TIC Rpt (H2 SO4)	TIC-D TIC-D	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54MW09		/	8-7-12	1025	H_2O	6		2	1	1	1	1		
54MW11		/	8-7-12	1140	H_2O	6		2	1	1	1	1		
54 [illegible] 8712		/	8-7-12	11:40	H_2O	2					1	1		
54 MW 08		/	8-7-12	12:45	H_2O	6		2	1	1	1	1		
54 MW 10		/	8-7-12	13:40	H_2O	6		2	1	1	1	1		
54MW B		/	8-7-12	1525	H_2O	6		2	1	1	1	1		
						32		10	5	5	6	6		

Relinquished by: (Signature)	Date	Time	Received by:
	8-7-12	1655	
Relinquished by: (Signature)	Date	Time	

Relinquished by: | Date | Time | Received by: (Signature)

Remarks:

Microbac OVD
Received: 08/08/2012 09:48
By: ROSEMARY SCOTT
221000027470

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

Generated: 08/22/2012 13:58

L12080279 / Revision: 0 / 535 total pages

COC No. A 31136

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: Shaw E&I

Project Contact: Eric Mahnke | Contact Phone #: 410-270-7233

Turn Around Requirements: STANDARD | Location: Radford

Project ID: SWMU 54 MNA

Sampler (print): William Groves | Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ AFCEE ☐ Other

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explo 8330	Aerobes (48 hrs)	Facultative (48 hrs)	Tic Fate (14 Sep)	Tic - D	Tic - D	TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS
54MW01			8-8-12	0740	H_2O	6		2	1	1	1	1			Chain has
54TM01			8-8-12	0740	H_2O	6		2	1	1	1	1			Two (2) coolers
54RB080812			8-8-12	0815	H_2O	6		2	1	1	1	1			Trackers #'s
54MW06			8-8-12	1205	H_2O	6		2	1	1	1	1			1Z401663220101285
54MW04			8-8-12	1330	H_2O	6		2	1	1	1	1			1Z401663220101285
MS8812			8-8-12	1330	H_2O	6		2	1	1	1	1			2334
MSD8812			8-8-12	1330	H_2O	6		2	1	1	1	1			
54MW07			8-8-12	1510	H_2O	6		2	1	1	1	1			
54MW14			8-8-12	1620	H_2O	6		2	1	1	1	1			
						59		18	9	9	9	9			

Relinquished by: (Signature)	Date	Time	Received by: (Signature)
	8-8-12	1650	
Relinquished by: (Signature)	Date	Time	Received by: (Signature)

Relinquished by: (Signature) | Date | Time | Received by: (Signature)

Remarks:

Microbac OVD
Received: 08/09/2012 09:43
By: ROSEMARY SCOTT
221000027505

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Generated: 08/22/2012 14:24

L12080318 / Revision: 0 / 459 total pages

COC No. A 31135

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name: Shaw E&I

Project Contact: Eric Malarek

Contact Phone #: 410-270-7233

Turn Around Requirements: Standard

Location: Canton

Project ID: 54MW54MNA

Sampler (print): Bill Groves

Signature:

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold	Explo 8330	Explosives (4 Metals)	Perchlorate	TNT/Tetr (HPLC)	TNT (GC-ECD)	TOTAL # (LAB USE)
54MW12		✓	8-9-12	0800	H_2O	6		2	1	1	1	1	
54TM12			8-9-12	0800	H_2O	6		2	1	1	1	1	
54MW03		✓	8-9-12	0900	H_2O	6		2	1	1	1	1	
54MW05		✓	8-9-12	1020	H_2O	6		2	1	1	1	1	
54MW02		✓	8-9-12	1210	H_2O	6		2	1	1	1	1	

Program
- ☐ CWA
- ☐ RCRA
- ☐ DOD
- ☐ AFCEE
- ☐ Other

ADDITIONAL REQUIREMENTS

Chain has Two (2) coolers
UPS Tracking #'s
1Z401663220101282611
1Z401663220101282711

Relinquished by: (Signature)	Date	Time
	8-9-12	1600
Relinquished by: (Signature)	Date	Time

Relinquished by:

Date	Time	Received by: (Signature)

Microbac OVD
Received: 08/10/2012 09:41
By: ROSEMARY SCOTT
221000027543

Remarks:

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page _____ of _____

Generated: 08/22/2012 14:24

Appendix B-2
Data Validation Reports

CB&I
4696 Millennium Drive, Suite 320
Belcamp, Maryland 21017
Tel: +1 (410) 273-7100
Fax: +1 (410) 273-7103
Eric.malarek@CBI.com

MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13050918

DATE: July 24, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 16, 2013. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of three aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW02	L13050918-01	54MW13	L13050918-05
54MW12	L13050918-03		

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
X		Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

7/24/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13050918

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/16/13, the cooler was received on 05/17/13 by the primary laboratory (Microbac Laboratories, Inc.) at 11.0°C. All samples were detected for perchlorate; therefore, were qualified estimated "L" for detections based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 05/16/13 for perchlorate analysis. The aqueous samples were prepped on 05/22/13 and analyzed on 05/22/13 and 05/23/13. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check
LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:
1- blank (<1/2MRL DoD Perchlorate Handbook)
5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
ICV (≤15%D DoD Perchlorate Handbook)
CCV/ICS (≤15%D DoD Perchlorate Handbook)
LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 03/04/13 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW02 (L13050918-01), 54MW12 (L13050918-03), and 54MW13 (L13050918-05) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
05/22/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
05/23/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
05/22/13	WG431309-02	All perchlorate <½MRL	NA	NA	None
05/22/13	RB051513	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG431309-03 was used as aqueous LCS for perchlorate analysis dated 05/22/13. All criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-01), 54MW12 (L13050918-03), and 54MW13 (L13050918-05) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- No site specific aqueous MS/MSD was performed for perchlorate analysis on 05/22/13; therefore, was not evaluated. See Section VII for lab accuracy statement using LCS.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No aqueous field duplicate was analyzed for perchlorate with this SDG; therefore, it was not evaluated.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW02 (L13050918-01), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.22
b = 0.000741
Y = Area = 130000/293000 = 0.444
X = 0.363

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.363 * 5 * 1) = 1.82 µg/L (Signal #1)

Reported Value = 1.82 µg/L
% Difference = 0.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050918-01
Client ID: 54MW02
Matrix: Water
Workgroup #: WG431309
Collect Date: 05/16/2013 07:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 05/22/2013 13:47
Cal Date: 03/04/2013 17:22
Run Date: 05/22/2013 21:59
File ID: 1LM.LM21183

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	1.82	L	0.200	0.100

Sample #: L13050918-03
Client ID: 54MW12
Matrix: Water
Workgroup #: WG431309
Collect Date: 05/16/2013 08:20
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 10
Units: ug/L

Instrument: LCMS1
Prep Date: 05/22/2013 13:47
Cal Date: 03/04/2013 17:22
Run Date: 05/23/2013 12:18
File ID: 1LM.LM21197

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	22.7	L	2.00	1.00

Sample #: L13050918-05
Client ID: 54MW13
Matrix: Water
Workgroup #: WG431309
Collect Date: 05/16/2013 09:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 05/22/2013 13:47
Cal Date: 03/04/2013 17:22
Run Date: 05/22/2013 23:53
File ID: 1LM.LM21189

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.640	L	0.200	0.100

CB&I
4696 Millennium Drive, Suite 320
Belcamp, Maryland 21017
Tel: +1 (410) 273-7100
Fax: +1 (410) 273-7103
Eric.malarek@CBIfederalservices.com

MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13050918

DATE: July 24, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 16, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of eight aqueous samples (includes five re-analysis samples) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW02	L13050918-01	54MW12	L13050918-03RE
54MW02	L13050918-01RE	54MW12	L13050918-03DLRE
54MW12	L13050918-03	54MW13	L13050918-05
54MW12	L13050918-03DL	54MW13	L13050918-05RE

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
X		Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications, except for the following. For samples 54MW12 (L13050918-03) and 54MW12 (L13050918-03RE), target compound 2,4,6-trinitrotoluene exceeded calibration range. Samples 54MW12 (L13050918-03DL) and 54MW12 (L13050918-03DLRE) should be used for this compound. There were no impacts for data usability based on this outlier.

Eric Malarek, Chemist

7/24/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13050918

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/16/13, the cooler was received on 05/17/13 by the primary laboratory (Microbac Laboratories, Inc.) at 11.0°C. All samples for explosives were qualified estimated "L" for detections and "UL" for non-detections based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 05/16/13. The samples were extracted on 05/22/13 and were analyzed on 05/23/13, 05/26/13, and 05/28/13 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
05/23/13	WG431232-01	All target explosives <½MRL	NA	NA	None
05/28/13	WG431232-01	All target explosives <½MRL	NA	NA	None
05/23/13	RB051513	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 03/20/13 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW02 (L13050918-01), 54MW12 (L13050918-03), 54MW12 (L13050918-03DL), and 54MW13 (L13050918-05) apply to this initial calibration.

- For the explosives initial calibration performed on 04/19/13 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Confirmation samples 54MW02 (L13050918-01RE), 54MW12 (L13050918-03RE), 54MW12 (L13050918-03DLRE), and 54MW13 (L13050918-05RE) apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 03/20/13 @17:40 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 05/23/13 @13:13 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-01) and 54MW12 (L13050918-03) were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/23/13 @22:00 on instrument HPLC5, all criteria were met. No qualifiers were applied. Sample 54MW13 (L13050918-05) was reported using this continuing calibration.

- For explosives continuing calibration performed on 05/24/13 @04:49 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/26/13 @20:48 on instrument HPLC5, all criteria were met. No qualifiers were applied. Sample 54MW12 (L13050918-03DL) was reported using this continuing calibration.

- For explosives continuing calibration performed on 05/27/13 @04:54 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 04/19/13 @17:22 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 05/28/13 @10:05 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation samples 54MW02 (L13050918-01RE), 54MW12 (L13050918-03RE), 54MW12 (L13050918-03DLRE), and 54MW13 (L13050918-05RE) were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/28/13 @19:05 on instrument HPLC4, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- For sample 54MW12 (L13050918-03DL), surrogate 1,2-dinitrobenzene was diluted out. No qualifiers were applied based upon this outlier.

- For sample 54MW12 (L13050918-03DLRE), surrogate 1,2-dinitrobenzene was diluted out. No qualifiers were applied based upon this outlier.

- For all other samples, all criteria were met. No qualifiers were applied.

VI-Laboratory Control Sample and Laboratory Control Sample Duplicate
Laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) are used to monitor long-term precision and accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Samples WG431232-02 and WG431232-03 were used as the aqueous LCS/LCSD for the explosives analysis on 05/23/13. All criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-01), 54MW12 (L13050918-03), 54MW12 (L13050918-03DL), and 54MW13 (L13050918-05) apply to this LCS/LCSD.

- Samples WG431232-02 and WG431232-03 were used as the aqueous LCS/LCSD for the explosives analysis on 05/28/13. All criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-01RE), 54MW12 (L13050918-03RE), 54MW12 (L13050918-03DLRE), and 54MW13 (L13050918-05RE) apply to this LCS/LCSD.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- No site specific aqueous MS/MSD was performed for explosives analysis on 05/23/13, 05/26/13, and 05/28/13; therefore, was not evaluated. See Section VI for lab accuracy and precision statements using LCS/LCSD.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- No aqueous field duplicate was analyzed for explosives with this SDG; therefore, it was not evaluated.

IX-Quantitation Verification and Data Review
The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- For samples 54MW12 (L13050918-03) and 54MW12 (L13050918-03RE), target compound 2,4,6-trinitrotoluene exceeded calibration range. Samples 54MW12 (L13050918-03DL) and 54MW12 (L13050918-03DLRE) should be used for this compound. There were no impacts for data usability based on this outlier.

- For sample 54MW12 (L13050918-03), target compound RDX (65.3%) was outside confirmation criteria; therefore, was qualified estimated based upon the high RPD.

- For sample 54MW12 (L13050918-03DL), target compounds 4-amino-2,6-dinitrotoluene (45.9%) and RDX (60.8%) were outside confirmation criteria; therefore, were qualified estimated based upon the high RPDs.

- For sample 54MW13 (L13050918-05), target compound 2-amino-4,6-dinitrotoluene (40.4%) was outside confirmation criteria; therefore, was qualified estimated based upon the high RPD.

- All other positive detections met confirmation criteria for all detected target analytes in this SDG.

Sample: 54MW02 (L13050918-01), 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.28909729
b = -1.8106455
Y = Area = 33.47058

X = 122.04

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (122.04 * 10 * 1) / (840) = 1.45 µg/L (Signal #1)

Reported Value = 1.45 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050918-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW02	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/23/2013 19:04
Collect Date:	05/16/2013 07:10	**Dilution:**	1	**File ID:**	5L009785.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U VL	1.19	0.298
2,4-Dinitrotoluene	121-14-2		U VL	1.19	0.298
2,6-Dinitrotoluene	606-20-2		U VL	1.19	0.298
2,4,6-Trinitrotoluene	118-96-7	0.749	J J	1.19	0.298
2-Amino-4,6-dinitrotoluene	35572-78-2	1.98	L	1.19	0.298
2-Nitrotoluene	88-72-2		U VL	1.19	0.298
4-Nitrotoluene	99-99-0		U VL	1.19	0.298
4-Amino-2,6-dinitrotoluene	19406-51-0	1.45	L	1.19	0.298
RDX	121-82-4		U VL	1.19	0.298
Nitroglycerin	55-63-0		U VL	1.19	0.298

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.0	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050918-01
Client ID: 54MW02
Matrix: Water
Workgroup #: WG431472
Collect Date: 05/16/2013 07:10
Sample Tag: CF01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC4
Prep Date: 05/22/2013 09:20
Cal Date: 04/19/2013 16:29
Run Date: 05/28/2013 13:38
File ID: 4L025173.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U VL	1.19	0.298
2,4-Dinitrotoluene	121-14-2		U VL	1.19	0.298
2,6-Dinitrotoluene	606-20-2		U VL	1.19	0.298
2,4,6-Trinitrotoluene	118-96-7	0.605	J J	1.19	0.298
2-Amino-4,6-dinitrotoluene	35572-78-2	1.67	L	1.19	0.298
2-Nitrotoluene	88-72-2		U VL	1.19	0.298
4-Nitrotoluene	99-99-0		U VL	1.19	0.298
4-Amino-2,6-dinitrotoluene	19406-51-0	1.26	L	1.19	0.298
RDX	121-82-4		U VL	1.19	0.298
Nitroglycerin	55-63-0		U VL	1.19	0.298

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	101	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050918-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW12	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	04/19/2013 16:29
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/28/2013 14:31
Collect Date:	05/16/2013 08:20	**Dilution:**	1	**File ID:**	4L025174.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U VL	1.16	0.291
2,4-Dinitrotoluene	121-14-2		U VL	1.16	0.291
2,6-Dinitrotoluene	606-20-2		U VL	1.16	0.291
2,4,6-Trinitrotoluene	118-96-7	110	J R	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2	7.94	L	1.16	0.291
2-Nitrotoluene	88-72-2		U VL	1.16	0.291
4-Nitrotoluene	99-99-0		U VL	1.16	0.291
4-Amino-2,6-dinitrotoluene	19406-51-0	4.71	L	1.16	0.291
RDX	121-82-4	12.7	J	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	67.2	50	150	

J	Estimated value ; the analyte concentration was greater than the highest standard
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050918-03
Client ID: 54MW12
Matrix: Water
Workgroup #: WG431472
Collect Date: 05/16/2013 08:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 05/22/2013 09:20
Cal Date: 03/20/2013 17:01
Run Date: 05/23/2013 19:43
File ID: 5L009786.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U VL	1.16	0.291
2,4-Dinitrotoluene	121-14-2		U VL	1.16	0.291
2,6-Dinitrotoluene	606-20-2		U VL	1.16	0.291
2,4,6-Trinitrotoluene	118-96-7	110	J R	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2	8.43	L	1.16	0.291
2-Nitrotoluene	88-72-2		U VL	1.16	0.291
4-Nitrotoluene	99-99-0		U VL	1.16	0.291
4-Amino-2,6-dinitrotoluene	19406-51-0	5.78	L	1.16	0.291
RDX	121-82-4	25.0	J	1.16	0.291
Nitroglycerin	55-63-0		U VL	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	64.8	50	150	

J	Estimated value ; the analyte concentration was greater than the highest standard
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050918-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW12	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	04/19/2013 16:29
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/28/2013 15:24
Collect Date:	05/16/2013 08:20	**Dilution:**	10	**File ID:**	4L025175.F
Sample Tag:	CFDL1	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U VL	11.6	2.91
2,4-Dinitrotoluene	121-14-2		U VL	11.6	2.91
2,6-Dinitrotoluene	606-20-2		U VL	11.6	2.91
2,4,6-Trinitrotoluene	118-96-7	106	L	11.6	2.91
2-Amino-4,6-dinitrotoluene	35572-78-2	7.34	L	11.6	2.91
2-Nitrotoluene	88-72-2		U VL	11.6	2.91
4-Nitrotoluene	99-99-0		U VL	11.6	2.91
4-Amino-2,6-dinitrotoluene	19406-51-0	3.79	J	11.6	2.91
RDX	121-82-4	12.6	J	11.6	2.91

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	DL	50	150	*

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050918-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW12	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/26/2013 21:27
Collect Date:	05/16/2013 08:20	**Dilution:**	10	**File ID:**	5L009837.F
Sample Tag:	DL01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U VL	11.6	2.91
2,4-Dinitrotoluene	121-14-2		U VL	11.6	2.91
2,6-Dinitrotoluene	606-20-2		U VL	11.6	2.91
2,4,6-Trinitrotoluene	118-96-7	108	L	11.6	2.91
2-Amino-4,6-dinitrotoluene	35572-78-2	8.80	L	11.6	2.91
2-Nitrotoluene	88-72-2		U VL	11.6	2.91
4-Nitrotoluene	99-99-0		U VL	11.6	2.91
4-Amino-2,6-dinitrotoluene	19406-51-0	6.05	J	11.6	2.91
RDX	121-82-4	23.6	J	11.6	2.91
Nitroglycerin	55-63-0		U VL	11.6	2.91

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	DL	50	150	*

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050918-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW13	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	04/19/2013 16:29
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/28/2013 16:17
Collect Date:	05/16/2013 09:35	**Dilution:**	1	**File ID:**	4L025176.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U VL	1.33	0.333
2,4-Dinitrotoluene	121-14-2		U VL	1.33	0.333
2,6-Dinitrotoluene	606-20-2		U VL	1.33	0.333
2,4,6-Trinitrotoluene	118-96-7	4.63	L	1.33	0.333
2-Amino-4,6-dinitrotoluene	35572-78-2	0.577	J J	1.33	0.333
2-Nitrotoluene	88-72-2		U VL	1.33	0.333
4-Nitrotoluene	99-99-0		U VL	1.33	0.333
4-Amino-2,6-dinitrotoluene	19406-51-0	0.628	J J	1.33	0.333
RDX	121-82-4	2.41	L	1.33	0.333
Nitroglycerin	55-63-0		U VL	1.33	0.333

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.6	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050918-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW13	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/23/2013 22:39
Collect Date:	05/16/2013 09:35	**Dilution:**	1	**File ID:**	5L009791.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U VL	1.33	0.333
2,4-Dinitrotoluene	121-14-2		U VL	1.33	0.333
2,6-Dinitrotoluene	606-20-2		U VL	1.33	0.333
2,4,6-Trinitrotoluene	118-96-7	4.91	L	1.33	0.333
2-Amino-4,6-dinitrotoluene	35572-78-2	0.869	J J	1.33	0.333
2-Nitrotoluene	88-72-2		U VL	1.33	0.333
4-Nitrotoluene	99-99-0		U VL	1.33	0.333
4-Amino-2,6-dinitrotoluene	19406-51-0	0.752	J J	1.33	0.333
RDX	121-82-4	3.20	L	1.33	0.333
Nitroglycerin	55-63-0		U VL	1.33	0.333

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	91.9	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13050918

DATE: July 24, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 16, 2013. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of three aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW02	L13050918-01	54MW13	L13050918-05
54MW12	L13050918-03		

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
X		Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

7/24/13
Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13050918

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/16/13, the cooler was received on 05/17/13 by the primary laboratory (Microbac Laboratories, Inc.) at 11.0°C. All samples were detected for chloride, sulfate, and nitrate; therefore, were qualified estimated "L" for detections based upon this outlier.

- Holding Time Review: Samples were collected on 05/16/13. The samples were prepped and analyzed on 05/17/13 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 04/10/13 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW02 (L13050918-01), 54MW12 (L13050918-03), and 54MW13 (L13050918-05) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
05/17/13	Chloride	ICB/CCBs	<LOD	NA	None
05/17/13	Sulfate	ICB/CCBs	<LOD	NA	None
05/17/13	Nitrate	ICB/CCBs	<LOD	NA	None
05/17/13	Chloride	WG430616-01	<½MRL	NA	None
05/17/13	Sulfate	WG430616-01	<½MRL	NA	None
05/17/13	Nitrate	WG430616-01	<½MRL	NA	None
05/17/13	Chloride	RB051513	0.451	2.26	None
05/17/13	Sulfate	RB051513	<½MRL	NA	None
05/17/13	Nitrate	RB051513	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG430616-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 05/17/13. All criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-01), 54MW12 (L13050918-03), and 54MW13 (L13050918-05) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- Sample 54MW13 (L13050918-05) was used as the aqueous MS/MSD for chloride, sulfate, and nitrate analysis on 05/17/13. All criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-01), 54MW12 (L13050918-03), and 54MW13 (L13050918-05) apply to this MS/MSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No aqueous field duplicate was analyzed for chloride, sulfate, and nitrate with this SDG; therefore, it was not evaluated.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW02 (L13050918-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1547
b = -0.4544
Y = Area = 3.274
DF = 1

X = 24.1 mg/L * DF = 24.1 mg/L * 1 = 24.1 mg/L

Reported concentration = 24.1 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050918-01
Client ID: 54MW02
Matrix: Water
Workgroup #: WG430616
Collect Date: 05/16/2013 07:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/17/2013 11:18
Cal Date: 04/10/2013 13:38
Run Date: 05/17/2013 16:32
File ID: I2_051713-21

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.46	L	0.200	0.100
Nitrate	14797-55-8	0.988	L	0.600	0.100
Sulfate	14808-79-8	24.1	L	1.00	0.500

Sample #: L13050918-03
Client ID: 54MW12
Matrix: Water
Workgroup #: WG430616
Collect Date: 05/16/2013 08:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/17/2013 11:18
Cal Date: 04/10/2013 13:38
Run Date: 05/17/2013 16:50
File ID: I2_051713-22

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.01	L	0.200	0.100
Nitrate	14797-55-8	4.03	L	0.600	0.100
Sulfate	14808-79-8	47.6	L	1.00	0.500

Sample #: L13050918-05
Client ID: 54MW13
Matrix: Water
Workgroup #: WG430616
Collect Date: 05/16/2013 09:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/17/2013 11:18
Cal Date: 04/10/2013 13:38
Run Date: 05/17/2013 17:07
File ID: I2_051713-23

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.90	L	0.200	0.100
Nitrate	14797-55-8	1.05	L	0.600	0.100
Sulfate	14808-79-8	29.2	L	1.00	0.500

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L13050819

DATE: July 19, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 14, 2013 and May 15, 2013. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of twenty four aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW03	L13050819-01	RB051513	L13050819-13
54MW03	L13050819-02	RB051513	L13050819-14
54MW06	L13050819-03	NRSW9/PW9	L13050819-15
54MW06	L13050819-04	NRSW9/PW9	L13050819-16
54MW09	L13050819-05	NRSW8/PW8	L13050819-17
54MW09	L13050819-06	NRSW8/PW8	L13050819-18
54MW11	L13050819-07	NRSW5/PW5	L13050819-19
54MW11	L13050819-08	NRSW5/PW5	L13050819-20
54MW08	L13050819-09	NRSW3/PW3	L13050819-21
54MW08	L13050819-10	NRSW3/PW3	L13050819-22
54MW10	L13050819-11	NRSW1/PW1	L13050819-23
54MW10	L13050819-12	NRSW1/PW1	L13050819-24

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

7/19/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13050819

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/14/13 and 05/15/13, the coolers were received on 05/16/13 by the primary laboratory (Microbac Laboratories, Inc.) at 6.0°C, 3.0°C, 3.0°C, and 5.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The samples were collected on 05/14/13 and 05/15/13. The TOC and TIC analysis were run on 05/23/13 and 05/28/13. The DOC and DIC analysis were run on 05/24/13. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC: 1 - blank
5 - standards ($r \geq 0.995$)
ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 05/23/13 and 05/28/13. The DOC and DIC analysis were run on 05/24/13. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW03 (L13050819-02), 54MW06 (L13050819-03), 54MW06 (L13050819-04), 54MW09 (L13050819-05), 54MW09 (L13050819-06), 54MW11 (L13050819-07), 54MW11 (L13050819-08), 54MW08 (L13050819-09), 54MW08 (L13050819-10), 54MW10 (L13050819-11), 54MW10 (L13050819-12), RB051513 (L13050819-13), RB051513 (L13050819-14), NRSW9/PW9 (L13050819-15), NRSW9/PW9 (L13050819-16), NRSW8/PW8 (L13050819-17), NRSW8/PW8 (L13050819-18), NRSW5/PW5 (L13050819-19), NRSW5/PW5 (L13050819-20), NRSW3/PW3 (L13050819-21), NRSW3/PW3 (L13050819-22), NRSW1/PW1 (L13050819-23), and NRSW1/PW1 (L13050819-24) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-14) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank RB051513 (L13050819-13) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
05/23/13	TOC	ICB/CCBs	<LOD	NA	None
05/28/13	TOC	ICB/CCBs	<LOD	NA	None
05/23/13	TOC	WG431487-01	<½MRL	NA	None
05/23/13	DOC	WG431489-01	<½MRL	NA	None
05/28/13	TOC	WG431760-01	<½MRL	NA	None
05/23/13	TOC	RB051513	0.514J	2.57	54MW03, 54MW06, 54MW09, 54MW11, NRSW9/PW9
05/23/13	TIC	RB051513	<½MRL	NA	None
05/24/13	DOC	RB051513	3.09	15.5	54MW03, 54MW06, 54MW09, 54MW11, 54MW08, 54MW10, NRSW9/PW9, NRSW8/PW8, NRSW5/PW5, NRSW3/PW3, NRSW1/PW1
05/24/13	DIC	RB051513	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG431487-02 and WG431487-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 05/23/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW3/PW3 (L13050819-21), and NRSW1/PW1 (L13050819-23) apply to this LCS/LCSD.

- Samples WG431760-02 and WG431760-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 05/28/13. All criteria were met. No qualifiers were applied. Sample NRSW5/PW5 (L13050819-19) applies to this LCS/LCSD.

- Samples WG431489-02 and WG431489-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 05/23/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-02), 54MW06 (L13050819-04), 54MW09 (L13050819-06), 54MW11 (L13050819-08), 54MW08 (L13050819-10), 54MW10 (L13050819-12), RB051513 (L13050819-14), NRSW9/PW9 (L13050819-16), NRSW8/PW8 (L13050819-18), NRSW5/PW5 (L13050819-20), NRSW3/PW3 (L13050819-22), and NRSW1/PW1 (L13050819-24) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- No site specific aqueous MS/MSD was performed for TOC and TIC analysis on 05/23/13 or 05/28/13; therefore, was not evaluated. See Section IV for lab accuracy statement using LCS/LCSD.

- No site specific aqueous MS/MSD was performed for DOC and DIC analysis on 05/23/13 or 05/24/13; therefore, was not evaluated. See Section IV for lab accuracy statement using LCS/LCSD.

VI-Laboratory Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statement using LCS/LCSD.

VII-Field Duplicate Sample Analysis

Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No aqueous field duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated.

VIII-Quantitation Verification and Data Review

The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW03 (L13050819-01), 54MW03 (L13050819-02), 54MW06 (L13050819-03), 54MW06 (L13050819-04), 54MW09 (L13050819-05), 54MW09 (L13050819-06), 54MW11 (L13050819-07), 54MW11 (L13050819-08), 54MW08 (L13050819-09), 54MW08 (L13050819-10), 54MW10 (L13050819-11), 54MW10 (L13050819-12), RB051513 (L13050819-13), RB051513 (L13050819-14), NRSW9/PW9 (L13050819-15), NRSW9/PW9 (L13050819-16), NRSW8/PW8 (L13050819-17), NRSW8/PW8 (L13050819-18), NRSW5/PW5 (L13050819-19), NRSW5/PW5 (L13050819-20), NRSW3/PW3 (L13050819-21), NRSW3/PW3 (L13050819-22), NRSW1/PW1 (L13050819-23), and NRSW1/PW1 (L13050819-24) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW03	TOC/DOC	1.0; 2.0	1.36	1.86J	26.9	J
54MW03	TIC/DIC	1.0; 2.0	33.3	55.7	40.2	J
54MW06	TOC/DOC	1.0; 2.0	2.41	2.24	NA	None
54MW06	TIC/DIC	1.0; 2.0	21.3	40.7	47.7	J
54MW09	TOC/DOC	1.0; 2.0	1.38	4.62	70.1	J
54MW09	TIC/DIC	1.0; 2.0	16.2	37.7	57.0	J
54MW11	TOC/DOC	1.0; 2.0	0.635J	2.76	77.0	J
54MW11	TIC/DIC	1.0; 2.0	20.7	53.8	61.5	J
54MW08	TOC/DOC	1.0; 2.0	1.00U	1.79J	NA	None
54MW08	TIC/DIC	1.0; 2.0	28.7	62.3	53.9	J
54MW10	TOC/DOC	1.0; 2.0	1.00U	2.34	NA	J, UJ
54MW10	TIC/DIC	1.0; 2.0	35.0	58.7	40.4	J
RB051513	TOC/DOC	1.0; 2.0	0.514J	3.09	83.4	J
RB051513	TIC/DIC	1.0; 2.0	1.00U	2.00U	NA	None
NRSW9/PW9	TOC/DOC	1.0; 2.0	0.810J	2.84	71.5	J
NRSW9/PW9	TIC/DIC	1.0; 2.0	21.9	34.4	36.3	J
NRSW8/PW8	TOC/DOC	1.0; 2.0	3.03	3.20	5.3	None
NRSW8/PW8	TIC/DIC	1.0; 2.0	23.8	50.4	52.8	J
NRSW5/PW5	TOC/DOC	2.0; 2.0	4.15	4.46	7.0	None
NRSW5/PW5	TIC/DIC	2.0; 2.0	32.6	86.6	62.4	J
NRSW3/PW3	TOC/DOC	1.0; 2.0	3.61	3.18	13.5	J
NRSW3/PW3	TIC/DIC	1.0; 2.0	8.27	18.4	55.1	J
NRSW1/PW1	TOC/DOC	1.0; 2.0	2.85	3.56	19.9	J
NRSW1/PW1	TIC/DIC	1.0; 2.0	4.78	12.7	62.4	J

J = Estimated value.
U = Non-detect as <MRL.
MRL = Method Reporting Limit
NA = Not Applicable

Sample: 54MW03 (L13050819-01), TOC

TC: Y = m*X (mg/L) + b	TIC: Y = m*X (mg/L) + b
m = 38.46	m = 29.16
b = 2.031	b = 6.703
Y = 1335	Y = 977.7
DF = 1	DF = 1
X = (34.66 mg/L) * 1 = 34.66 mg/L	X = (33.30 mg/L) * 1 = 33.30 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 34.66 – 33.30 = 1.36 mg/L

Reported Value = 1.36 mg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW03	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 19:04
Collect Date:	05/14/2013 16:45	**Dilution:**	1	**File ID:**	TC05232013.011
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.36	B	1.00	0.500

Sample #:	L13050819-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW03	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 19:04
Collect Date:	05/14/2013 16:45	**Dilution:**	1	**File ID:**	TC05232013.011
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		33.3	J	1.00	0.500

Sample #:	L13050819-02	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW03	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 00:08
Collect Date:	05/14/2013 16:45	**Dilution:**	2	**File ID:**	TC05232013.035
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.86	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Certificate of Analysis

Sample #: L13050819-02 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW03 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431489 **Analyst:** BAF **Run Date:** 05/24/2013 00:08
Collect Date: 05/14/2013 16:45 **Dilution:** 2 **File ID:** TC05232013.035
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		55.7	J	2.00	1.00

Sample #: L13050819-03 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW06 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431487 **Analyst:** BAF **Run Date:** 05/23/2013 19:18
Collect Date: 05/14/2013 18:10 **Dilution:** 1 **File ID:** TC05232013.012
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.41	B	1.00	0.500

Sample #: L13050819-03 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW06 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431487 **Analyst:** BAF **Run Date:** 05/23/2013 19:18
Collect Date: 05/14/2013 18:10 **Dilution:** 1 **File ID:** TC05232013.012
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		21.3	J	1.00	0.500

Sample #: L13050819-04 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW06 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431489 **Analyst:** BAF **Run Date:** 05/24/2013 00:22
Collect Date: 05/14/2013 18:10 **Dilution:** 2 **File ID:** TC05232013.036
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.24	B	2.00	1.00

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050819-04
Client ID: 54MW06
Matrix: Water
Workgroup #: WG431489
Collect Date: 05/14/2013 18:10
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/24/2013 00:22
File ID: TC05232013.036

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		40.7	J	2.00	1.00

Sample #: L13050819-05
Client ID: 54MW09
Matrix: Water
Workgroup #: WG431487
Collect Date: 05/15/2013 08:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/23/2013 19:31
File ID: TC05232013.013

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		16.2	J	1.00	0.500

Sample #: L13050819-05
Client ID: 54MW09
Matrix: Water
Workgroup #: WG431487
Collect Date: 05/15/2013 08:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/23/2013 19:31
File ID: TC05232013.013

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.38	B	1.00	0.500

Sample #: L13050819-06
Client ID: 54MW09
Matrix: Water
Workgroup #: WG431489
Collect Date: 05/15/2013 08:25
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/24/2013 00:35
File ID: TC05232013.037

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		37.7	J	2.00	1.00

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-06	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW09	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 00:35
Collect Date:	05/15/2013 08:25	**Dilution:**	2	**File ID:**	TC05232013.037
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.62	B	2.00	1.00

Sample #:	L13050819-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW11	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:05
Collect Date:	05/15/2013 09:35	**Dilution:**	1	**File ID:**	TC05232013.016
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		20.7	J	1.00	0.500

Sample #:	L13050819-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW11	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:05
Collect Date:	05/15/2013 09:35	**Dilution:**	1	**File ID:**	TC05232013.016
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.635	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Sample #:	L13050819-08	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW11	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 01:10
Collect Date:	05/15/2013 09:35	**Dilution:**	2	**File ID:**	TC05232013.040
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.76	B	2.00	1.00

Sample #:	L13050819-08	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW11	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 01:10
Collect Date:	05/15/2013 09:35	**Dilution:**	2	**File ID:**	TC05232013.040
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		53.8	J	2.00	1.00

Sample #:	L13050819-09	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW08	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:19
Collect Date:	05/15/2013 10:45	**Dilution:**	1	**File ID:**	TC05232013.017
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		28.7	J	1.00	0.500

Sample #:	L13050819-09	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW08	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:19
Collect Date:	05/15/2013 10:45	**Dilution:**	1	**File ID:**	TC05232013.017
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC		U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-10	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW08	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 01:23
Collect Date:	05/15/2013 10:45	**Dilution:**	2	**File ID:**	TC05232013.041
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		62.3	J	2.00	1.00

Sample #:	L13050819-10	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW08	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 01:23
Collect Date:	05/15/2013 10:45	**Dilution:**	2	**File ID:**	TC05232013.041
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.79	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-11	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:32
Collect Date:	05/15/2013 12:05	**Dilution:**	1	**File ID:**	TC05232013.018
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC		U UJ	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Sample #:	L13050819-11	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:32
Collect Date:	05/15/2013 12:05	**Dilution:**	1	**File ID:**	TC05232013.018
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		35.0	J	1.00	0.500

Sample #:	L13050819-12	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 01:37
Collect Date:	05/15/2013 12:05	**Dilution:**	2	**File ID:**	TC05232013.042
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.34	B	2.00	1.00

Sample #:	L13050819-12	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 01:37
Collect Date:	05/15/2013 12:05	**Dilution:**	2	**File ID:**	TC05232013.042
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		58.7	J	2.00	1.00

Sample #:	L13050819-13	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	RB051513	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:45
Collect Date:	05/15/2013 12:35	**Dilution:**	1	**File ID:**	TC05232013.019
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.514	J J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-13	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	RB051513	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:45
Collect Date:	05/15/2013 12:35	**Dilution:**	1	**File ID:**	TC05232013.019
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-14	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	RB051513	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 01:50
Collect Date:	05/15/2013 12:35	**Dilution:**	2	**File ID:**	TC05232013.043
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.09	J	2.00	1.00

Sample #:	L13050819-14	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	RB051513	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 01:50
Collect Date:	05/15/2013 12:35	**Dilution:**	2	**File ID:**	TC05232013.043
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved			U	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-15	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW9/PW9	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:57
Collect Date:	05/15/2013 12:45	**Dilution:**	1	**File ID:**	TC05232013.020
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		21.9	J	1.00	0.500

Sample #:	L13050819-15	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW9/PW9	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 20:57
Collect Date:	05/15/2013 12:45	**Dilution:**	1	**File ID:**	TC05232013.020
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.810	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-16	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW9/PW9	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 02:02
Collect Date:	05/15/2013 12:45	**Dilution:**	2	**File ID:**	TC05232013.044
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.84	B	2.00	1.00

Sample #:	L13050819-16	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW9/PW9	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 02:02
Collect Date:	05/15/2013 12:45	**Dilution:**	2	**File ID:**	TC05232013.044
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		34.4	J	2.00	1.00

Sample #:	L13050819-17	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW8/PW8	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 21:11
Collect Date:	05/15/2013 13:20	**Dilution:**	1	**File ID:**	TC05232013.021
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		23.8	J	1.00	0.500

Sample #:	L13050819-17	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW8/PW8	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 21:11
Collect Date:	05/15/2013 13:20	**Dilution:**	1	**File ID:**	TC05232013.021
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.03		1.00	0.500

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-18	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW8/PW8	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 02:15
Collect Date:	05/15/2013 13:20	**Dilution:**	2	**File ID:**	TC05232013.045
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		50.4	J	2.00	1.00

Sample #:	L13050819-18	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW8/PW8	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 02:15
Collect Date:	05/15/2013 13:20	**Dilution:**	2	**File ID:**	TC05232013.045
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.20	B	2.00	1.00

Sample #:	L13050819-19	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW5/PW5	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431760	**Analyst:**	BAF	**Run Date:**	05/28/2013 11:43
Collect Date:	05/15/2013 13:50	**Dilution:**	2	**File ID:**	TC05282013.006
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		32.6	J	2.00	1.00

Sample #:	L13050819-19	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW5/PW5	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431760	**Analyst:**	BAF	**Run Date:**	05/28/2013 11:43
Collect Date:	05/15/2013 13:50	**Dilution:**	2	**File ID:**	TC05282013.006
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	4.15		2.00	1.00

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-20	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW5/PW5	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 09:05
Collect Date:	05/15/2013 13:50	**Dilution:**	2	**File ID:**	TC05232013.076
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.46	B	2.00	1.00

Sample #:	L13050819-20	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW5/PW5	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 09:05
Collect Date:	05/15/2013 13:50	**Dilution:**	2	**File ID:**	TC05232013.076
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		86.6	J	2.00	1.00

Sample #:	L13050819-21	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW3/PW3	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 21:39
Collect Date:	05/15/2013 14:35	**Dilution:**	1	**File ID:**	TC05232013.023
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.61	J	1.00	0.500

Sample #:	L13050819-21	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW3/PW3	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431487	**Analyst:**	BAF	**Run Date:**	05/23/2013 21:39
Collect Date:	05/15/2013 14:35	**Dilution:**	1	**File ID:**	TC05232013.023
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		8.27	J	1.00	0.500

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Certificate of Analysis

Sample #: L13050819-22 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: NRSW3/PW3 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431489 **Analyst:** BAF **Run Date:** 05/24/2013 02:29
Collect Date: 05/15/2013 14:35 **Dilution:** 2 **File ID:** TC05232013.046
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		18.4	J	2.00	1.00

Sample #: L13050819-22 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: NRSW3/PW3 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431489 **Analyst:** BAF **Run Date:** 05/24/2013 02:29
Collect Date: 05/15/2013 14:35 **Dilution:** 2 **File ID:** TC05232013.046
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.18	B	2.00	1.00

Sample #: L13050819-23 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: NRSW1/PW1 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431487 **Analyst:** BAF **Run Date:** 05/23/2013 21:52
Collect Date: 05/15/2013 15:30 **Dilution:** 1 **File ID:** TC05232013.024
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		4.78	J	1.00	0.500

Sample #: L13050819-23 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: NRSW1/PW1 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431487 **Analyst:** BAF **Run Date:** 05/23/2013 21:52
Collect Date: 05/15/2013 15:30 **Dilution:** 1 **File ID:** TC05232013.024
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.85	J	1.00	0.500

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-24	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW1/PW1	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 02:41
Collect Date:	05/15/2013 15:30	**Dilution:**	2	**File ID:**	TC05232013.047
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.56	B	2.00	1.00

Sample #:	L13050819-24	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	NRSW1/PW1	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431489	**Analyst:**	BAF	**Run Date:**	05/24/2013 02:41
Collect Date:	05/15/2013 15:30	**Dilution:**	2	**File ID:**	TC05232013.047
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		12.7	J	2.00	1.00

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13050819

DATE: July 19, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 14, 2013 and May 15, 2013. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of twelve aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW03	L13050819-01	RB051513	L13050819-13
54MW06	L13050819-03	NRSW9/PW9	L13050819-15
54MW09	L13050819-05	NRSW8/PW8	L13050819-17
54MW11	L13050819-07	NRSW5/PW5	L13050819-19
54MW08	L13050819-09	NRSW3/PW3	L13050819-21
54MW10	L13050819-11	NRSW1/PW1	L13050819-23

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

7/19/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13050819

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/14/13 and 05/15/13, the coolers were received on 05/16/13 by the primary laboratory (Microbac Laboratories, Inc.) at 6.0°C, 3.0°C, 3.0°C, and 5.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 05/14/13 and 05/15/13 for perchlorate analysis. The aqueous samples were prepped and analyzed on 05/22/13. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check
LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:
1- blank (<1/2MRL DoD Perchlorate Handbook)
5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
ICV (≤15%D DoD Perchlorate Handbook)
CCV/ICS (≤15%D DoD Perchlorate Handbook)
LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 03/04/13 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW03 (L13050819-01), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), NRSW1/PW1 (L13050819-23) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
05/21/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
05/22/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
05/22/13	WG431309-02	All perchlorate <½MRL	NA	NA	None
05/22/13	RB051513	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG431309-03 was used as aqueous LCS for perchlorate analysis dated 05/22/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), NRSW1/PW1 (L13050819-23) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- No site specific aqueous MS/MSD was performed for perchlorate analysis on 05/22/13; therefore, was not evaluated. See Section VII for lab accuracy statement using LCS.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No aqueous field duplicate was analyzed for perchlorate with this SDG; therefore, it was not evaluated.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW03 (L13050819-01), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.22
b = 0.000741
Y = Area = 49800/346000 = 0.144
X = 0.1174

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.1174 * 5 * 1) = 0.587 µg/L (Signal #1)

Reported Value = 0.590 µg/L
% Difference = 0.5%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-01	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW03	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 16:56
Collect Date:	05/14/2013 16:45	**Dilution:**	1	**File ID:**	1LM.LM21167
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.590		0.200	0.100

Sample #:	L13050819-03	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW06	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 17:15
Collect Date:	05/14/2013 18:10	**Dilution:**	1	**File ID:**	1LM.LM21168
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.171	J J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

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Certificate of Analysis

Sample #: L13050819-05 **PrePrep Method:** N/A **Instrument:** LCMS1
Client ID: 54MW09 **Prep Method:** 6850 **Prep Date:** 05/22/2013 13:47
Matrix: Water **Analytical Method:** 6850 **Cal Date:** 03/04/2013 17:22
Workgroup #: WG431309 **Analyst:** JWR **Run Date:** 05/22/2013 17:34
Collect Date: 05/15/2013 08:25 **Dilution:** 1 **File ID:** 1LM.LM21169
Sample Tag: 01 **Units:** ug/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.923		0.200	0.100

Sample #: L13050819-07 **PrePrep Method:** N/A **Instrument:** LCMS1
Client ID: 54MW11 **Prep Method:** 6850 **Prep Date:** 05/22/2013 13:47
Matrix: Water **Analytical Method:** 6850 **Cal Date:** 03/04/2013 17:22
Workgroup #: WG431309 **Analyst:** JWR **Run Date:** 05/22/2013 19:09
Collect Date: 05/15/2013 09:35 **Dilution:** 1 **File ID:** 1LM.LM21174
Sample Tag: 01 **Units:** ug/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.430		0.200	0.100

Sample #: L13050819-09 **PrePrep Method:** N/A **Instrument:** LCMS1
Client ID: 54MW08 **Prep Method:** 6850 **Prep Date:** 05/22/2013 13:47
Matrix: Water **Analytical Method:** 6850 **Cal Date:** 03/04/2013 17:22
Workgroup #: WG431309 **Analyst:** JWR **Run Date:** 05/22/2013 19:28
Collect Date: 05/15/2013 10:45 **Dilution:** 1 **File ID:** 1LM.LM21175
Sample Tag: 01 **Units:** ug/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.118	J J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-11	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW10	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 19:47
Collect Date:	05/15/2013 12:05	**Dilution:**	1	**File ID:**	1LM.LM21176
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.835		0.200	0.100

Sample #:	L13050819-13	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	RB051513	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 20:06
Collect Date:	05/15/2013 12:35	**Dilution:**	1	**File ID:**	1LM.LM21177
Sample Tag:	01	**Units:**	ug/L		

Analyte		CAS #	Result	Qual	LOQ	LOD
Perchlorate		14797-73-0		U	0.200	0.100
U	Analyte was not detected. The concentration is below the reported LOD.					

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-15	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	NRSW9/PW9	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 20:24
Collect Date:	05/15/2013 12:45	**Dilution:**	1	**File ID:**	1LM.LM21178
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-17	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	NRSW8/PW8	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 20:43
Collect Date:	05/15/2013 13:20	**Dilution:**	1	**File ID:**	1LM.LM21179
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-19	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	NRSW5/PW5	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 21:02
Collect Date:	05/15/2013 13:50	**Dilution:**	1	**File ID:**	1LM.LM21180
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-21	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	NRSW3/PW3	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 21:21
Collect Date:	05/15/2013 14:35	**Dilution:**	1	**File ID:**	1LM.LM21181
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-23	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	NRSW1/PW1	**Prep Method:**	6850	**Prep Date:**	05/22/2013 13:47
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431309	**Analyst:**	JWR	**Run Date:**	05/22/2013 21:40
Collect Date:	05/15/2013 15:30	**Dilution:**	1	**File ID:**	1LM.LM21182
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13050819

DATE: July 22, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 14, 2013 and May 15, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of fourteen aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW03	L13050819-01	54MW10	L13050819-11RE
54MW03	L13050819-01RE	RB051513	L13050819-13
54MW06	L13050819-03	NRSW9/PW9	L13050819-15
54MW09	L13050819-05	NRSW8/PW8	L13050819-17
54MW11	L13050819-07	NRSW5/PW5	L13050819-19
54MW08	L13050819-09	NRSW3/PW3	L13050819-21
54MW10	L13050819-11	NRSW1/PW1	L13050819-23

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

7/22/13
Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13050819

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/14/13 and 05/15/13, the coolers were received on 05/16/13 by the primary laboratory (Microbac Laboratories, Inc.) at 6.0°C, 3.0°C, 3.0°C, and 5.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 05/14/13 and 05/15/13. The samples were extracted on 05/21/13 and 05/22/13 and were analyzed on 05/23/13 and 05/28/13 for explosives, respectively. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
05/22/13	WG431046-01	All target explosives <½MRL	NA	NA	None
05/23/13	WG431232-01	All target explosives <½MRL	NA	NA	None
05/27/13	WG431046-01	All target explosives <½MRL	NA	NA	None
05/23/13	RB051513	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 03/20/13 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), and NRSW1/PW1 (L13050819-23) apply to this initial calibration.

- For the explosives initial calibration performed on 04/19/13 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Confirmation samples 54MW03 (L13050819-01RE) and 54MW10 (L13050819-11RE) apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 03/20/13 @17:40 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 05/22/13 @10:49 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/22/13 @20:05 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW06 (L13050819-03), and 54MW09 (L13050819-05) were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/23/13 @04:52 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), and NRSW8/PW8 (L13050819-17) were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/23/13 @11:02 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/23/13 @13:13 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), and NRSW1/PW1 (L13050819-23) were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/23/13 @22:00 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 04/19/13 @17:22 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 05/27/13 @14:20 on instrument HPLC4, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/28/13 @02:52 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation samples 54MW03 (L13050819-01RE) and 54MW10 (L13050819-11RE) were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/28/13 @10:05 on instrument HPLC4, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met. No qualifiers were applied.

VI-Laboratory Control Sample and Laboratory Control Sample Duplicate
Laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) are used to monitor long-term precision and accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Sample WG431046-02 was used as the aqueous LCS for the explosives analysis on 05/22/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW03 (L13050819-01RE), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), 54MW10 (L13050819-11RE), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), NRSW1/PW1 (L13050819-23) apply to this LCS.

- Samples WG431232-02 and WG431232-03 were used as the aqueous LCS/LCSD for the explosives analysis on 05/23/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW03 (L13050819-01RE), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), 54MW10 (L13050819-11RE), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), NRSW1/PW1 (L13050819-23) apply to this LCS/LCSD.

- Sample WG431046-02 was used as the aqueous LCS for the explosives analysis on 05/27/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW03 (L13050819-01RE), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), 54MW10 (L13050819-11RE), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), NRSW1/PW1 (L13050819-23) apply to this LCS.

VII-Matrix Spike/Matrix Spike Duplicate

Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- No site specific aqueous MS/MSD was performed for explosives analysis on 05/22/13, 05/23/13, and 05/27/13; therefore, was not evaluated. See Section VI for lab accuracy and precision statements using LCS/LCSD.

VIII-Field Duplicate Sample Analysis

Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- No aqueous field duplicate was analyzed for explosives with this SDG; therefore, it was not evaluated.

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- For sample 54MW10 (L13050819-11), target compound 4-amino-2,6-dinitrotoluene (62.8%) was outside confirmation criteria; therefore, was qualified estimated based upon the high RPD.

- All other positive detections met confirmation criteria for all detected target analytes in this SDG.

Sample: 54MW03 (L13050819-01), 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.28909729
b = -1.8106455
Y = Area = 9.75521

X = 40.007

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (40.007 * 10 * 1) / (880) = 0.455 µg/L (Signal #1)

Reported Value = 0.455 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Microbac

Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW03	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 01:17
Collect Date:	05/14/2013 16:45	**Dilution:**	1	**File ID:**	5L009758.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0	0.455	J J	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	103	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW03	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	04/19/2013 16:29
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/28/2013 04:39
Collect Date:	05/14/2013 16:45	**Dilution:**	1	**File ID:**	4L025162.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0	0.318	J J	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	112	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW06	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 01:56
Collect Date:	05/14/2013 18:10	**Dilution:**	1	**File ID:**	5L009759.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.09	0.272
2,4-Dinitrotoluene	121-14-2		U	1.09	0.272
2,6-Dinitrotoluene	606-20-2		U	1.09	0.272
2,4,6-Trinitrotoluene	118-96-7		U	1.09	0.272
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.09	0.272
2-Nitrotoluene	88-72-2		U	1.09	0.272
4-Nitrotoluene	99-99-0		U	1.09	0.272
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.09	0.272
RDX	121-82-4		U	1.09	0.272
Nitroglycerin	55-63-0		U	1.09	0.272

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	88.8	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW09	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 02:35
Collect Date:	05/15/2013 08:25	**Dilution:**	1	**File ID:**	5L009760.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.16	0.291
2,4-Dinitrotoluene	121-14-2		U	1.16	0.291
2,6-Dinitrotoluene	606-20-2		U	1.16	0.291
2,4,6-Trinitrotoluene	118-96-7		U	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.16	0.291
2-Nitrotoluene	88-72-2		U	1.16	0.291
4-Nitrotoluene	99-99-0		U	1.16	0.291
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.16	0.291
RDX	121-82-4		U	1.16	0.291
Nitroglycerin	55-63-0		U	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	85.0	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-07	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW11	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 05:31
Collect Date:	05/15/2013 09:35	**Dilution:**	1	**File ID:**	5L009765.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.16	0.291
2,4-Dinitrotoluene	121-14-2		U	1.16	0.291
2,6-Dinitrotoluene	606-20-2		U	1.16	0.291
2,4,6-Trinitrotoluene	118-96-7		U	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.16	0.291
2-Nitrotoluene	88-72-2		U	1.16	0.291
4-Nitrotoluene	99-99-0		U	1.16	0.291
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.16	0.291
RDX	121-82-4		U	1.16	0.291
Nitroglycerin	55-63-0		U	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	106	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW08	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 06:10
Collect Date:	05/15/2013 10:45	**Dilution:**	1	**File ID:**	5L009766.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	105	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-11	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW10	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 06:49
Collect Date:	05/15/2013 12:05	**Dilution:**	1	**File ID:**	5L009767.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7	0.637	J J	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2	0.652	J J	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0	0.877	J J	1.14	0.284
RDX	121-82-4	1.29		1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	103	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-11	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW10	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	04/19/2013 16:29
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/28/2013 06:25
Collect Date:	05/15/2013 12:05	**Dilution:**	1	**File ID:**	4L025164.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7	0.573	J J	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2	0.451	J J	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0	0.458	J J	1.14	0.284
RDX	121-82-4	1.37		1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	109	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-13	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	RB051513	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 07:28
Collect Date:	05/15/2013 12:35	**Dilution:**	1	**File ID:**	5L009768.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.16	0.291
2,4-Dinitrotoluene	121-14-2		U	1.16	0.291
2,6-Dinitrotoluene	606-20-2		U	1.16	0.291
2,4,6-Trinitrotoluene	118-96-7		U	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.16	0.291
2-Nitrotoluene	88-72-2		U	1.16	0.291
4-Nitrotoluene	99-99-0		U	1.16	0.291
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.16	0.291
RDX	121-82-4		U	1.16	0.291
Nitroglycerin	55-63-0		U	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	95.8	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-15	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	NRSW9/PW9	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 08:07
Collect Date:	05/15/2013 12:45	**Dilution:**	1	**File ID:**	5L009769.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	0.581	0.145
2,4-Dinitrotoluene	121-14-2		U	0.581	0.145
2,6-Dinitrotoluene	606-20-2		U	0.581	0.145
2,4,6-Trinitrotoluene	118-96-7		U	0.581	0.145
2-Amino-4,6-dinitrotoluene	35572-78-2		U	0.581	0.145
2-Nitrotoluene	88-72-2		U	0.581	0.145
4-Nitrotoluene	99-99-0		U	0.581	0.145
4-Amino-2,6-dinitrotoluene	19406-51-0		U	0.581	0.145
RDX	121-82-4		U	0.581	0.145
Nitroglycerin	55-63-0		U	0.581	0.145

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	101	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-17	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	NRSW8/PW8	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/23/2013 08:46
Collect Date:	05/15/2013 13:20	**Dilution:**	1	**File ID:**	5L009770.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	0.568	0.142
2,4-Dinitrotoluene	121-14-2		U	0.568	0.142
2,6-Dinitrotoluene	606-20-2		U	0.568	0.142
2,4,6-Trinitrotoluene	118-96-7		U	0.568	0.142
2-Amino-4,6-dinitrotoluene	35572-78-2		U	0.568	0.142
2-Nitrotoluene	88-72-2		U	0.568	0.142
4-Nitrotoluene	99-99-0		U	0.568	0.142
4-Amino-2,6-dinitrotoluene	19406-51-0		U	0.568	0.142
RDX	121-82-4		U	0.568	0.142
Nitroglycerin	55-63-0		U	0.568	0.142

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	92.8	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-19	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	NRSW5/PW5	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/23/2013 17:07
Collect Date:	05/15/2013 13:50	**Dilution:**	1	**File ID:**	5L009782.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	106	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-21	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	NRSW3/PW3	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/23/2013 17:46
Collect Date:	05/15/2013 14:35	**Dilution:**	1	**File ID:**	5L009783.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	100	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-23	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	NRSW1/PW1	**Prep Method:**	3535	**Prep Date:**	05/22/2013 09:20
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431472	**Analyst:**	ECL	**Run Date:**	05/23/2013 18:25
Collect Date:	05/15/2013 15:30	**Dilution:**	1	**File ID:**	5L009784.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.61	0.403
2,4-Dinitrotoluene	121-14-2		U	1.61	0.403
2,6-Dinitrotoluene	606-20-2		U	1.61	0.403
2,4,6-Trinitrotoluene	118-96-7		U	1.61	0.403
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.61	0.403
2-Nitrotoluene	88-72-2		U	1.61	0.403
4-Nitrotoluene	99-99-0		U	1.61	0.403
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.61	0.403
RDX	121-82-4		U	1.61	0.403
Nitroglycerin	55-63-0		U	1.61	0.403

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	105	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13050819

DATE: July 19, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 14, 2013 and May 15, 2013. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of twelve aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW03	L13050819-01	RB051513	L13050819-13
54MW06	L13050819-03	NRSW9/PW9	L13050819-15
54MW09	L13050819-05	NRSW8/PW8	L13050819-17
54MW11	L13050819-07	NRSW5/PW5	L13050819-19
54MW08	L13050819-09	NRSW3/PW3	L13050819-21
54MW10	L13050819-11	NRSW1/PW1	L13050819-23

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

7/19/13
Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13050819

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/14/13 and 05/15/13, the coolers were received on 05/16/13 by the primary laboratory (Microbac Laboratories, Inc.) at 6.0°C, 3.0°C, 3.0°C, and 5.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: Samples were collected on 05/14/13 and 05/15/13. The samples were prepped and analyzed on 05/17/13 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 04/10/13 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), NRSW1/PW1 (L13050819-23) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
05/17/13	Chloride	ICB/CCBs	<LOD	NA	None
05/17/13	Sulfate	ICB/CCBs	<LOD	NA	None
05/17/13	Nitrate	ICB/CCBs	<LOD	NA	None
05/17/13	Chloride	WG430616-01	<½MRL	NA	None
05/17/13	Sulfate	WG430616-01	<½MRL	NA	None
05/17/13	Nitrate	WG430616-01	<½MRL	NA	None
05/17/13	Chloride	RB051513	0.451	2.26	54MW06
05/17/13	Sulfate	RB051513	<½MRL	NA	None
05/17/13	Nitrate	RB051513	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG430616-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 05/17/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), NRSW1/PW1 (L13050819-23) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- Sample 54MW13 (L13050918-05) was used as the aqueous MS/MSD for chloride, sulfate, and nitrate analysis on 05/17/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13050819-01), 54MW06 (L13050819-03), 54MW09 (L13050819-05), 54MW11 (L13050819-07), 54MW08 (L13050819-09), 54MW10 (L13050819-11), RB051513 (L13050819-13), NRSW9/PW9 (L13050819-15), NRSW8/PW8 (L13050819-17), NRSW5/PW5 (L13050819-19), NRSW3/PW3 (L13050819-21), NRSW1/PW1 (L13050819-23) apply to this MS/MSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No aqueous field duplicate was analyzed for chloride, sulfate, and nitrate with this SDG; therefore, it was not evaluated.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW03 (L13050819-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1547
b = -0.4544
Y = Area = 4.032
DF = 1

X = 29.0 mg/L * DF = 29.0 mg/L * 1 = 29.0 mg/L

Reported concentration = 29.0 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-01	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW03	**Prep Method:**	9056	**Prep Date:**	05/17/2013 11:18
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430616	**Analyst:**	JBK	**Run Date:**	05/17/2013 12:28
Collect Date:	05/14/2013 16:45	**Dilution:**	1	**File ID:**	I2_051713-07
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.45		0.200	0.100
Nitrate	14797-55-8	0.911		0.600	0.100
Sulfate	14808-79-8	29.0		1.00	0.500

Sample #:	L13050819-03	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW06	**Prep Method:**	9056	**Prep Date:**	05/17/2013 11:18
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430616	**Analyst:**	JBK	**Run Date:**	05/17/2013 12:45
Collect Date:	05/14/2013 18:10	**Dilution:**	1	**File ID:**	I2_051713-08
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.24	B	0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	14.0		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050819-05
Client ID: 54MW09
Matrix: Water
Workgroup #: WG430616
Collect Date: 05/15/2013 08:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/17/2013 11:18
Cal Date: 04/10/2013 13:38
Run Date: 05/17/2013 13:03
File ID: I2_051713-09

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.80		0.200	0.100
Nitrate	14797-55-8	0.637		0.600	0.100
Sulfate	14808-79-8	96.7		1.00	0.500

Sample #: L13050819-07
Client ID: 54MW11
Matrix: Water
Workgroup #: WG430616
Collect Date: 05/15/2013 09:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/17/2013 11:18
Cal Date: 04/10/2013 13:38
Run Date: 05/17/2013 13:20
File ID: I2_051713-10

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.99		0.200	0.100
Nitrate	14797-55-8	0.647		0.600	0.100
Sulfate	14808-79-8	89.4		1.00	0.500

Sample #: L13050819-09
Client ID: 54MW08
Matrix: Water
Workgroup #: WG430616
Collect Date: 05/15/2013 10:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/17/2013 11:18
Cal Date: 04/10/2013 13:38
Run Date: 05/17/2013 13:38
File ID: I2_051713-11

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.73		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	41.1		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050819-11 **PrePrep Method:** N/A **Instrument:** IC2
Client ID: 54MW10 **Prep Method:** 9056 **Prep Date:** 05/17/2013 11:18
Matrix: Water **Analytical Method:** 9056 **Cal Date:** 04/10/2013 13:38
Workgroup #: WG430616 **Analyst:** JBK **Run Date:** 05/17/2013 13:55
Collect Date: 05/15/2013 12:05 **Dilution:** 1 **File ID:** I2_051713-12
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.04		0.200	0.100
Nitrate	14797-55-8	0.740		0.600	0.100
Sulfate	14808-79-8	48.8		1.00	0.500

Sample #: L13050819-13 **PrePrep Method:** N/A **Instrument:** IC2
Client ID: RB051513 **Prep Method:** 9056 **Prep Date:** 05/17/2013 11:18
Matrix: Water **Analytical Method:** 9056 **Cal Date:** 04/10/2013 13:38
Workgroup #: WG430616 **Analyst:** JBK **Run Date:** 05/17/2013 14:13
Collect Date: 05/15/2013 12:35 **Dilution:** 1 **File ID:** I2_051713-13
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	0.451		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8		U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

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Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-15	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	NRSW9/PW9	**Prep Method:**	9056	**Prep Date:**	05/17/2013 11:18
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430616	**Analyst:**	JBK	**Run Date:**	05/17/2013 14:30
Collect Date:	05/15/2013 12:45	**Dilution:**	1	**File ID:**	I2_051713-14
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	7.70		0.200	0.100
Nitrate	14797-55-8	0.461	J J	0.600	0.100
Sulfate	14808-79-8	18.1		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Microbac

Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-17	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	NRSW8/PW8	**Prep Method:**	9056	**Prep Date:**	05/17/2013 11:18
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430616	**Analyst:**	JBK	**Run Date:**	05/17/2013 15:22
Collect Date:	05/15/2013 13:20	**Dilution:**	1	**File ID:**	I2_051713-17
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	7.47		0.200	0.100
Nitrate	14797-55-8	0.393	J J	0.600	0.100
Sulfate	14808-79-8	7.62		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-19	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	NRSW5/PW5	**Prep Method:**	9056	**Prep Date:**	05/17/2013 11:18
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430616	**Analyst:**	JBK	**Run Date:**	05/17/2013 15:40
Collect Date:	05/15/2013 13:50	**Dilution:**	1	**File ID:**	I2_051713-18
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	10.4		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	9.50		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L13050819
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050819-21	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	NRSW3/PW3	**Prep Method:**	9056	**Prep Date:**	05/17/2013 11:18
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430616	**Analyst:**	JBK	**Run Date:**	05/17/2013 15:57
Collect Date:	05/15/2013 14:35	**Dilution:**	1	**File ID:**	I2_051713-19
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	6.86		0.200	0.100
Nitrate	14797-55-8	0.723		0.600	0.100
Sulfate	14808-79-8	7.20		1.00	0.500

Sample #:	L13050819-23	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	NRSW1/PW1	**Prep Method:**	9056	**Prep Date:**	05/17/2013 11:18
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430616	**Analyst:**	JBK	**Run Date:**	05/17/2013 16:15
Collect Date:	05/15/2013 15:30	**Dilution:**	1	**File ID:**	I2_051713-20
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	6.38		0.200	0.100
Nitrate	14797-55-8	0.642		0.600	0.100
Sulfate	14808-79-8	7.15		1.00	0.500

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L13050733

DATE: July 18, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 14, 2013. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of fourteen aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW04	L13050733-01	54MW14	L13050733-08
54MW04	L13050733-02	54MW05	L13050733-09
54TMW01	L13050733-03	54MW05	L13050733-10
54TMW01	L13050733-04	54TMW02	L13050733-11
54MW07	L13050733-05	54TMW02	L13050733-12
54MW07	L13050733-06	54MW01	L13050733-13
54MW14	L13050733-07	54MW01	L13050733-14

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
X		Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
X		Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

7/18/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13050733

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/14/13, the coolers were received on 05/15/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C, 3.0°C, and 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The samples were collected on 05/14/13. The TOC and TIC analysis were run on 05/21/13. The DOC and DIC analysis were run on 05/21/13. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC: 1 - blank
5 - standards ($r \geq 0.995$)
ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 05/21/13. The DOC and DIC analysis were run on 05/21/13. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54MW04 (L13050733-02), 54TMW01 (L13050733-03), 54TMW01 (L13050733-04), 54MW07 (L13050733-05), 54MW07 (L13050733-06), 54MW14 (L13050733-07), 54MW14 (L13050733-08), 54MW05 (L13050733-09), 54MW05 (L13050733-10), 54TMW02 (L13050733-11), 54TMW02 (L13050733-12), 54MW01 (L13050733-13), and 54MW01 (L13050733-14) apply to these initial and continuing calibrations.

III-Blank Analysis
The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-14) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank RB051513 (L13050819-13) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
02/22/13	TOC	ICB/CCBs	<LOD	NA	None
02/22/13	TOC	WG430883-01	<½MRL	NA	None
02/25/13	DOC	WG430881-01	0.733	3.67	54MW04, 54MW07, 54MW14, 54TMW02
05/23/13	TOC	RB051513	0.514J	2.57	54MW04, 54MW07, 54MW14, 54MW05, 54TMW02, 54MW01
05/23/13	TIC	RB051513	<½MRL	NA	None
05/24/13	DOC	RB051513	3.09	15.5	54MW04, 54TMW01, 54MW07, 54MW14, 54MW05, 54TMW02, 54MW01
05/24/13	DIC	RB051513	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG430883-02 and WG430883-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 05/21/13. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this LCS/LCSD.

- Samples WG430881-02 and WG430881-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 05/21/13. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-02), 54TMW01 (L13050733-04), 54MW07 (L13050733-06), 54MW14 (L13050733-08), 54MW05 (L13050733-10), 54TMW02 (L13050733-12), and 54MW01 (L13050733-14) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- Sample 54MW01 (L13050733-13) was used as the aqueous MS/MSD for TOC and TIC analysis on 05/21/13. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this MS/MSD.

- Sample 54MW01 (L13050733-14) was used as the aqueous MS/MSD for DOC and DIC analysis on 05/21/13. DOC (77.3%, 75.9%) was outside criteria. All criteria were met for DIC. For DOC, all associated samples were qualified estimated bias low "L" for detections and no qualification for non-detections based upon the high recovery. Samples 54MW04 (L13050733-02), 54TMW01 (L13050733-04), 54MW07 (L13050733-06), 54MW14 (L13050733-08), 54MW05 (L13050733-10), 54TMW02 (L13050733-12), and 54MW01 (L13050733-14) apply to this MS/MSD.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW04 (L13050733-01) and 54TMW01 (L13050733-03) was collected for TOC and TIC analysis. TOC was detected at 0.619J mg/L in the parent sample and non-detect at <1.00 mg/L in the duplicate pair. No qualifiers were applied based upon this outlier. TIC was detected at 5.85 mg/L in the parent sample and at 6.68 mg/L in the duplicate pair resulting in a RPD of 13.3% (within limits). All criteria were met. No qualifiers were applied.

- Field aqueous sample duplicate pair 54MW04 (L13050733-02) and 54TMW01 (L13050733-04) was collected for DOC and DIC analysis. DOC was detected at 2.74 mg/L in the parent sample and at 3.89 mg/L in the duplicate pair resulting in a RPD of 34.7% (outside limits). DIC was detected at 52.5 mg/L in the parent sample and at 52.3 mg/L in the duplicate pair resulting in a RPD of 0.4% (within limits). All criteria were met for DIC. DOC was outside limits; therefore, was qualified estimated "J" for the duplicate pair based upon the high RPD.

- Field aqueous sample duplicate pair 54MW05 (L13050733-09) and 54TMW02 (L13050733-11) was collected for TOC and TIC analysis. TOC was detected at 1.06 mg/L in the parent sample and at 1.10 mg/L in the duplicate pair resulting in a RPD of 3.7% (within limits). TIC was detected at 5.34 mg/L in the parent sample and at 4.34 mg/L in the duplicate pair resulting in a RPD of 13.3% (within limits). All criteria were met. No qualifiers were applied.

- Field aqueous sample duplicate pair 54MW05 (L13050733-10) and 54TMW02 (L13050733-12) was collected for DOC and DIC analysis. DOC was detected at 4.06 mg/L in the parent sample and at 1.65 mg/L in the duplicate pair resulting in a RPD of 84.4% (outside limits). DIC was detected at 45.8 mg/L in the parent sample and at 46.2 mg/L in the duplicate pair resulting in a RPD of 0.9% (within limits). All criteria were met for DIC. DOC was outside limits; therefore, was qualified estimated "J" for the duplicate pair based upon the high RPD.

VIII-Quantitation Verification and Data Review

The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW04 (L13050733-01), 54MW04 (L13050733-02), 54TMW01 (L13050733-03), 54TMW01 (L13050733-04), 54MW07 (L13050733-05), 54MW07 (L13050733-06), 54MW14 (L13050733-07), 54MW14 (L13050733-08), 54MW05 (L13050733-09), 54MW05 (L13050733-10), 54TMW02 (L13050733-11), 54TMW02 (L13050733-12), 54MW01 (L13050733-13), and 54MW01 (L13050733-14) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW04	TOC/DOC	1.0; 2.0	0.619J	2.74	77.4	J
54MW04	TIC/DIC	1.0; 2.0	5.85	52.5	88.9	J
54TMW01	TOC/DOC	1.0; 2.0	1.00U	3.89	NA	J, UJ
54TMW01	TIC/DIC	1.0; 2.0	6.68	52.3	87.2	J
54MW07	TOC/DOC	1.0; 2.0	0.793J	1.93J	58.9	J
54MW07	TIC/DIC	1.0; 2.0	5.42	57.0	90.5	J
54MW14	TOC/DOC	1.0; 2.0	0.583J	3.01	80.6	J
54MW14	TIC/DIC	1.0; 2.0	7.14	67.5	89.4	J
54MW05	TOC/DOC	1.0; 2.0	1.06	4.06	73.9	J
54MW05	TIC/DIC	1.0; 2.0	5.34	45.8	88.3	J
54TMW02	TOC/DOC	1.0; 1.0	1.10	1.65	33.3	J
54TMW02	TIC/DIC	1.0; 1.0	4.34	46.2	90.6	J
54MW01	TOC/DOC	1.0; 2.0	0.949J	4.34	78.1	J
54MW01	TIC/DIC	1.0; 2.0	3.08	42.6	92.8	J

J = Estimated value.
U = Non-detect as <MRL.
MRL = Method Reporting Limit
NA = Not Applicable

Sample: 54MW04 (L13050733-01), TOC

TC: Y = m*X (mg/L) + b	TIC: Y = m*X (mg/L) + b
m = 38.46	m = 29.16
b = 2.031	b = 6.703
Y = 250.7	Y = 177.2
DF = 1	DF = 1
X = (6.47 mg/L) * 1 = 6.47 mg/L	X = (5.85 mg/L) * 1 = 5.85 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 6.47 – 5.85 = 0.62 mg/L

Reported Value = 0.619 mg/L
% Difference = 0.2%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050733-01
Client ID: 54MW04
Matrix: Water
Workgroup #: WG430883
Collect Date: 05/14/2013 08:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 04:58
File ID: TC05202013.057

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		5.85	J	1.00	0.500

Sample #: L13050733-01
Client ID: 54MW04
Matrix: Water
Workgroup #: WG430883
Collect Date: 05/14/2013 08:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 04:58
File ID: TC05202013.057

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.619	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Certificate of Analysis

Sample #: L13050733-02 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW04 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430881 **Analyst:** BAF **Run Date:** 05/21/2013 01:46
Collect Date: 05/14/2013 08:55 **Dilution:** 2 **File ID:** TC05202013.042
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.74	B	2.00	1.00

Sample #: L13050733-02 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW04 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430881 **Analyst:** BAF **Run Date:** 05/21/2013 01:46
Collect Date: 05/14/2013 08:55 **Dilution:** 2 **File ID:** TC05202013.042
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		52.5	J	2.00	1.00

Sample #: L13050733-03 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54TMW01 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430883 **Analyst:** BAF **Run Date:** 05/21/2013 05:10
Collect Date: 05/14/2013 08:55 **Dilution:** 1 **File ID:** TC05202013.058
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC		U UJ	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Certificate of Analysis

Sample #: L13050733-03 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54TMW01 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430883 **Analyst:** BAF **Run Date:** 05/21/2013 05:10
Collect Date: 05/14/2013 08:55 **Dilution:** 1 **File ID:** TC05202013.058
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		6.68	J	1.00	0.500

Sample #: L13050733-04 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54TMW01 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430881 **Analyst:** BAF **Run Date:** 05/21/2013 11:57
Collect Date: 05/14/2013 08:55 **Dilution:** 2 **File ID:** TC05202013.078
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.89	B	2.00	1.00

Sample #: L13050733-04 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54TMW01 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430881 **Analyst:** BAF **Run Date:** 05/21/2013 11:57
Collect Date: 05/14/2013 08:55 **Dilution:** 2 **File ID:** TC05202013.078
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		52.3	J	2.00	1.00

Sample #: L13050733-05 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW07 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430883 **Analyst:** BAF **Run Date:** 05/21/2013 05:23
Collect Date: 05/14/2013 10:15 **Dilution:** 1 **File ID:** TC05202013.059
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		5.42	J	1.00	0.500

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-05	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW07	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG430883	**Analyst:**	BAF	**Run Date:**	05/21/2013 05:23
Collect Date:	05/14/2013 10:15	**Dilution:**	1	**File ID:**	TC05202013.059
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.793	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050733-06
Client ID: 54MW07
Matrix: Water
Workgroup #: WG430881
Collect Date: 05/14/2013 10:15
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 12:10
File ID: TC05202013.079

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		57.0	J	2.00	1.00

Sample #: L13050733-06
Client ID: 54MW07
Matrix: Water
Workgroup #: WG430881
Collect Date: 05/14/2013 10:15
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 12:10
File ID: TC05202013.079

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.93	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW14	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG430883	**Analyst:**	BAF	**Run Date:**	05/21/2013 05:35
Collect Date:	05/14/2013 11:20	**Dilution:**	1	**File ID:**	TC05202013.060
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		7.14	J	1.00	0.500

Sample #:	L13050733-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW14	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG430883	**Analyst:**	BAF	**Run Date:**	05/21/2013 05:35
Collect Date:	05/14/2013 11:20	**Dilution:**	1	**File ID:**	TC05202013.060
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.583	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Certificate of Analysis

Sample #: L13050733-08
Client ID: 54MW14
Matrix: Water
Workgroup #: WG430881
Collect Date: 05/14/2013 11:20
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 02:29
File ID: TC05202013.045

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		67.5	J	2.00	1.00

Sample #: L13050733-08
Client ID: 54MW14
Matrix: Water
Workgroup #: WG430881
Collect Date: 05/14/2013 11:20
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 02:29
File ID: TC05202013.045

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.01	B	2.00	1.00

Sample #: L13050733-09
Client ID: 54MW05
Matrix: Water
Workgroup #: WG430883
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 05:48
File ID: TC05202013.061

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.06	B	1.00	0.500

Sample #: L13050733-09
Client ID: 54MW05
Matrix: Water
Workgroup #: WG430883
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 05:48
File ID: TC05202013.061

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		5.34	J	1.00	0.500

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Certificate of Analysis

Sample #: L13050733-10
Client ID: 54MW05
Matrix: Water
Workgroup #: WG430881
Collect Date: 05/14/2013 12:30
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 02:43
File ID: TC05202013.046

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.06	B	2.00	1.00

Sample #: L13050733-10
Client ID: 54MW05
Matrix: Water
Workgroup #: WG430881
Collect Date: 05/14/2013 12:30
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 02:43
File ID: TC05202013.046

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		45.8	J	2.00	1.00

Sample #: L13050733-11
Client ID: 54TMW02
Matrix: Water
Workgroup #: WG430883
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 06:21
File ID: TC05202013.064

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		4.34	J	1.00	0.500

Sample #: L13050733-11
Client ID: 54TMW02
Matrix: Water
Workgroup #: WG430883
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 06:21
File ID: TC05202013.064

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.10	B	1.00	0.500

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Microbac

Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050733-12
Client ID: 54TMW02
Matrix: Water
Workgroup #: WG430881
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 02:56
File ID: TC05202013.047

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		46.2	J	1.00	0.500

Sample #: L13050733-12
Client ID: 54TMW02
Matrix: Water
Workgroup #: WG430881
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 02:56
File ID: TC05202013.047

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.65	B	1.00	0.500

Sample #: L13050733-13
Client ID: 54MW01
Matrix: Water
Workgroup #: WG430883
Collect Date: 05/14/2013 13:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/21/2013 06:34
File ID: TC05202013.065

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.949	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Certificate of Analysis

Sample #: L13050733-13 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW01 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430883 **Analyst:** BAF **Run Date:** 05/21/2013 06:34
Collect Date: 05/14/2013 13:55 **Dilution:** 1 **File ID:** TC05202013.065
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		3.08	J	1.00	0.500

Sample #: L13050733-14 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW01 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430881 **Analyst:** BAF **Run Date:** 05/21/2013 03:10
Collect Date: 05/14/2013 13:55 **Dilution:** 2 **File ID:** TC05202013.048
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.34	B	2.00	1.00

Sample #: L13050733-14 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW01 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430881 **Analyst:** BAF **Run Date:** 05/21/2013 03:10
Collect Date: 05/14/2013 13:55 **Dilution:** 2 **File ID:** TC05202013.048
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		42.6	J	2.00	1.00

Sample #: L13050733-15 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW01 MS **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG430883 **Analyst:** BAF **Run Date:** 05/21/2013 06:46
Collect Date: 05/14/2013 13:55 **Dilution:** 1 **File ID:** TC05202013.066
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		0.972	J J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13050733

DATE: June 21, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 14, 2013. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of seven aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW04	L13050733-01	54MW05	L13050733-09
54TMW01	L13050733-03	54TMW02	L13050733-11
54MW07	L13050733-05	54MW01	L13050733-13
54MW14	L13050733-07		

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/21/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13050733

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/14/13, the coolers were received on 05/15/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C, 3.0°C, and 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 05/14/13 for perchlorate analysis. The aqueous samples were prepped on 05/21/13 and analyzed on 05/21/13 and 05/22/13. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check
LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:
1- blank (<1/2MRL DoD Perchlorate Handbook)
5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
ICV (≤15%D DoD Perchlorate Handbook)
CCV/ICS (≤15%D DoD Perchlorate Handbook)
LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 03/04/13 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
05/21/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
05/22/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
05/21/13	WG431115-02	All perchlorate <½MRL	NA	NA	None
05/22/13	RB051513	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG431115-03 was used as aqueous LCS for perchlorate analysis dated 05/21/13. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- Sample 54MW01 (L13050733-13) was used as the aqueous MS/MSD for the perchlorate analysis on 05/21/13. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this MS/MSD.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW04 (L13050733-01) and 54TMW01 (L13050733-03) was analyzed for perchlorate analysis in this SDG. Perchlorate was non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

- Field groundwater sample duplicate pair 54MW05 (L13050733-09) and 54TMW02 (L13050733-11) was analyzed for perchlorate analysis in this SDG. Perchlorate was detected at 0.309 µg/L in the parent sample and at 0.321 µg/L in the duplicate pair; resulting in a RPD of 3.8%. All criteria were met. No qualifiers were applied.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW07 (L13050733-05), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.22
b = 0.000741
Y = Area = 8560/333000 = 0.025706
X = 0.02046

Conc. μg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in μg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (μg/L)
DF = Dilution factor

Conc. μg/L = (0.02046 * 5 * 1) = 0.102 μg/L (Signal #1)

Reported Value = 0.103 μg/L
% Difference = 1.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-01	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW04	**Prep Method:**	6850	**Prep Date:**	05/21/2013 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431115	**Analyst:**	JWR	**Run Date:**	05/21/2013 22:37
Collect Date:	05/14/2013 08:55	**Dilution:**	1	**File ID:**	1LM.LM21137
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-03	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54TMW01	**Prep Method:**	6850	**Prep Date:**	05/21/2013 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431115	**Analyst:**	JWR	**Run Date:**	05/21/2013 22:56
Collect Date:	05/14/2013 08:55	**Dilution:**	1	**File ID:**	1LM.LM21138
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-05	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW07	**Prep Method:**	6850	**Prep Date:**	05/21/2013 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431115	**Analyst:**	JWR	**Run Date:**	05/21/2013 23:15
Collect Date:	05/14/2013 10:15	**Dilution:**	1	**File ID:**	1LM.LM21139
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.103	J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-07	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW14	**Prep Method:**	6850	**Prep Date:**	05/21/2013 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	03/04/2013 17:22
Workgroup #:	WG431115	**Analyst:**	JWR	**Run Date:**	05/21/2013 23:34
Collect Date:	05/14/2013 11:20	**Dilution:**	1	**File ID:**	1LM.LM21140
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.195	J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

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Certificate of Analysis

Sample #: L13050733-09
Client ID: 54MW05
Matrix: Water
Workgroup #: WG431115
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 05/21/2013 16:00
Cal Date: 03/04/2013 17:22
Run Date: 05/21/2013 23:53
File ID: 1LM.LM21141

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.309		0.200	0.100

Sample #: L13050733-11
Client ID: 54TMW02
Matrix: Water
Workgroup #: WG431115
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 05/21/2013 16:00
Cal Date: 03/04/2013 17:22
Run Date: 05/22/2013 00:12
File ID: 1LM.LM21142

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.321		0.200	0.100

Sample #: L13050733-13
Client ID: 54MW01
Matrix: Water
Workgroup #: WG431115
Collect Date: 05/14/2013 13:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 05/21/2013 16:00
Cal Date: 03/04/2013 17:22
Run Date: 05/22/2013 01:46
File ID: 1LM.LM21147

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13050733

DATE: June 21, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 14, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of seven aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW04	L13050733-01	54MW05	L13050733-09
54TMW01	L13050733-03	54TMW02	L13050733-11
54MW07	L13050733-05	54MW01	L13050733-13
54MW14	L13050733-07		

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
X		System Monitoring Compounds
	X	Laboratory Control Sample
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/21/13
Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13050733

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/14/13, the coolers were received on 05/15/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C, 3.0°C, and 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 05/14/13. The samples were extracted on 05/21/13 and were analyzed on 05/22/13 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
05/22/13	WG431046-01	All target explosives <½MRL	NA	NA	None
05/23/13	RB051513	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 03/20/13 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this initial calibration.

- For the explosives initial calibration performed on 04/19/13 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. No confirmation samples were required for all associated samples were non-detect. No samples apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 03/20/13 @17:40 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 05/22/13 @10:49 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), and 54MW01 (L13050733-13) were reported using this continuing calibration.

- For explosives continuing calibration performed on 05/22/13 @20:05 on instrument HPLC5, all criteria were met. No qualifiers were applied. Sample 54TMW02 (L13050733-11) was reported using this continuing calibration.

- For explosives continuing calibration performed on 05/23/13 @04:52 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- For sample 54TM01 (L13020607-05), surrogate 1,2-dinitrobenzene (26.4%) was below criteria. All target compounds were non-detect for the sample; therefore, were qualified estimated "UJ" based upon the low recovery.

- For all other samples, all criteria were met.

VI-Laboratory Control Sample
Laboratory control samples (LCS) are used to monitor long-term accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used.

- Sample WG431046-02 was used as the aqueous LCS for the explosives analysis on 05/22/13. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this LCS.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Sample 54MW01 (L13050733-13) was used as the aqueous MS/MSD for the explosives analysis on 05/22/13. Target compounds 1,3-dinitrobenzene (RPD=20.1%), 2,4-dinitrotoluene (RPD=22.1%), 2,6-dinitrotoluene (RPD=21.2%), 2-nitrotoluene (RPD=22.0%), and 4-nitrotoluene (RPD=22.5%) were outside precision criteria. All recovery criteria were met. All associated samples were non-detect for all target explosives; therefore, no qualifiers were applied based upon the high RPDs. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this MS/MSD.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW04 (L13050733-01) and 54TMW01 (L13050733-03) was analyzed for explosives analysis in this SDG. All target explosives were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

- Field groundwater sample duplicate pair 54MW05 (L13050733-09) and 54TMW02 (L13050733-11) was analyzed for explosives analysis in this SDG. All target explosives were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

IX-Quantitation Verification and Data Review
The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- All associated samples were non-detect for all target compounds. No further confirmation criterion is required in this SDG.

Sample: WG431046-02, 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.28909729
b = -1.8106455
Y = Area = 119.10315

X = 418.25

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (418.25 * 10 * 1) / (1000) = 4.18 µg/L (Signal #1)

Reported Value = 4.18 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

Form I Copy

Microbac

Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050733-01
Client ID: 54MW04
Matrix: Water
Workgroup #: WG431218
Collect Date: 05/14/2013 08:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 05/21/2013 10:00
Cal Date: 03/20/2013 17:01
Run Date: 05/22/2013 13:15
File ID: 5L009739.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	99.5	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54TMW01	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/22/2013 13:54
Collect Date:	05/14/2013 08:55	**Dilution:**	1	**File ID:**	5L009740.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.16	0.291
2,4-Dinitrotoluene	121-14-2		U	1.16	0.291
2,6-Dinitrotoluene	606-20-2		U	1.16	0.291
2,4,6-Trinitrotoluene	118-96-7		U	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.16	0.291
2-Nitrotoluene	88-72-2		U	1.16	0.291
4-Nitrotoluene	99-99-0		U	1.16	0.291
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.16	0.291
RDX	121-82-4		U	1.16	0.291
Nitroglycerin	55-63-0		U	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	93.5	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW07	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/22/2013 14:33
Collect Date:	05/14/2013 10:15	**Dilution:**	1	**File ID:**	5L009741.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		UJ UJ	8.33	2.08
2,4-Dinitrotoluene	121-14-2		UJ UJ	8.33	2.08
2,6-Dinitrotoluene	606-20-2		UJ UJ	8.33	2.08
2,4,6-Trinitrotoluene	118-96-7		UJ UJ	8.33	2.08
2-Amino-4,6-dinitrotoluene	35572-78-2		UJ UJ	8.33	2.08
2-Nitrotoluene	88-72-2		UJ UJ	8.33	2.08
4-Nitrotoluene	99-99-0		UJ UJ	8.33	2.08
4-Amino-2,6-dinitrotoluene	19406-51-0		UJ UJ	8.33	2.08
RDX	121-82-4		UJ UJ	8.33	2.08
Nitroglycerin	55-63-0		UJ UJ	8.33	2.08

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	26.4	50	150	*

UJ	Undetected; the analyte was analyzed for, but not detected.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-07	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW14	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/22/2013 15:12
Collect Date:	05/14/2013 11:20	**Dilution:**	1	**File ID:**	5L009742.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.16	0.291
2,4-Dinitrotoluene	121-14-2		U	1.16	0.291
2,6-Dinitrotoluene	606-20-2		U	1.16	0.291
2,4,6-Trinitrotoluene	118-96-7		U	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.16	0.291
2-Nitrotoluene	88-72-2		U	1.16	0.291
4-Nitrotoluene	99-99-0		U	1.16	0.291
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.16	0.291
RDX	121-82-4		U	1.16	0.291
Nitroglycerin	55-63-0		U	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	98.5	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW05	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/22/2013 15:51
Collect Date:	05/14/2013 12:30	**Dilution:**	1	**File ID:**	5L009743.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	88.6	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050733-11
Client ID: 54TMW02
Matrix: Water
Workgroup #: WG431218
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 05/21/2013 10:00
Cal Date: 03/20/2013 17:01
Run Date: 05/22/2013 20:44
File ID: 5L009751.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.19	0.298
2,4-Dinitrotoluene	121-14-2		U	1.19	0.298
2,6-Dinitrotoluene	606-20-2		U	1.19	0.298
2,4,6-Trinitrotoluene	118-96-7		U	1.19	0.298
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.19	0.298
2-Nitrotoluene	88-72-2		U	1.19	0.298
4-Nitrotoluene	99-99-0		U	1.19	0.298
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.19	0.298
RDX	121-82-4		U	1.19	0.298
Nitroglycerin	55-63-0		U	1.19	0.298

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	92.3	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-13	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW01	**Prep Method:**	3535	**Prep Date:**	05/21/2013 10:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	03/20/2013 17:01
Workgroup #:	WG431218	**Analyst:**	ECL	**Run Date:**	05/22/2013 16:31
Collect Date:	05/14/2013 13:55	**Dilution:**	1	**File ID:**	5L009744.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	99.2	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Eric.malarek@CBIfederalservices.com

MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13050733

DATE: July 18, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 14, 2013. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of seven aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW04	L13050733-01	54MW05	L13050733-09
54TMW01	L13050733-03	54TMW02	L13050733-11
54MW07	L13050733-05	54MW01	L13050733-13
54MW14	L13050733-07		

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

E. Malarek

Eric Malarek, Chemist

7/18/13

Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13050733

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/14/13, the coolers were received on 05/15/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C, 3.0°C, and 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: Samples were collected on 05/14/13. The samples were prepped and analyzed on 05/16/13 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 04/10/13 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-13) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
05/16/13	Chloride	ICB/CCBs	<LOD	NA	None
05/16/13	Sulfate	ICB/CCBs	<LOD	NA	None
05/16/13	Nitrate	ICB/CCBs	<LOD	NA	None
05/16/13	Chloride	WG430447-01	<½MRL	NA	None
05/16/13	Sulfate	WG430447-01	<½MRL	NA	None
05/16/13	Nitrate	WG430447-01	<½MRL	NA	None
05/17/13	Chloride	RB051513	0.451	2.26	54MW01
05/17/13	Sulfate	RB051513	<½MRL	NA	None
05/17/13	Nitrate	RB051513	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG430447-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 05/16/13. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate

Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at

a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- Sample 54MW01 (L13050733-13) was used as the aqueous MS/MSD for chloride, sulfate, and nitrate analysis on 05/16/13. All criteria were met. No qualifiers were applied. Samples 54MW04 (L13050733-01), 54TMW01 (L13050733-03), 54MW07 (L13050733-05), 54MW14 (L13050733-07), 54MW05 (L13050733-09), 54TMW02 (L13050733-11), and 54MW01 (L13050733-13) apply to this MS/MSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW04 (L13050733-01) and 54TMW01 (L13050733-03) was analyzed for chloride, sulfate, and nitrate analysis in this SDG. Chloride was detected at 3.24 mg/L in the parent sample and at 3.18 mg/L in the duplicate pair; resulting in a RPD of 1.9%. Sulfate was detected at 329 mg/L in the parent sample and at 331 mg/L in the duplicate pair; resulting in a RPD of 0.6%. Nitrate was non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

- Field groundwater sample duplicate pair 54MW05 (L13050733-09) and 54TMW02 (L13050733-11) was analyzed for chloride, sulfate, and nitrate analysis in this SDG. Chloride was detected at 5.51 mg/L in the parent sample and at 5.57 mg/L in the duplicate pair; resulting in a RPD of 1.1%. Sulfate was detected at 15.7 mg/L in the parent sample and at 15.6 mg/L in the duplicate pair; resulting in a RPD of 0.6%. Nitrate was detected at 1.60 mg/L in the parent sample and at 1.60 mg/L in the duplicate pair; resulting in a RPD of 0.0%. All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW04 (L13050733-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1547
b = -0.4544
Y = Area = 9.717
DF = 5

X = 65.75 mg/L * DF = 65.75 mg/L * 5 = 329 mg/L

Reported concentration = 329 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-01	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW04	**Prep Method:**	9056	**Prep Date:**	05/16/2013 11:25
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430447	**Analyst:**	JBK	**Run Date:**	05/16/2013 13:24
Collect Date:	05/14/2013 08:55	**Dilution:**	5	**File ID:**	I2_051613-07
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.24		1.00	0.500
Nitrate	14797-55-8		U	3.00	0.500
Sulfate	14808-79-8	329		5.00	2.50

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-03	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54TMW01	**Prep Method:**	9056	**Prep Date:**	05/16/2013 11:25
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430447	**Analyst:**	JBK	**Run Date:**	05/16/2013 13:43
Collect Date:	05/14/2013 08:55	**Dilution:**	5	**File ID:**	I2_051613-08
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.18		1.00	0.500
Nitrate	14797-55-8		U	3.00	0.500
Sulfate	14808-79-8	331		5.00	2.50

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-05	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW07	**Prep Method:**	9056	**Prep Date:**	05/16/2013 11:25
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430447	**Analyst:**	JBK	**Run Date:**	05/16/2013 14:01
Collect Date:	05/14/2013 10:15	**Dilution:**	3	**File ID:**	I2_051613-09
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.64		0.600	0.300
Nitrate	14797-55-8	1.10	J J	1.80	0.300
Sulfate	14808-79-8	137		3.00	1.50

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050733-07	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW14	**Prep Method:**	9056	**Prep Date:**	05/16/2013 11:25
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/10/2013 13:38
Workgroup #:	WG430447	**Analyst:**	JBK	**Run Date:**	05/16/2013 14:20
Collect Date:	05/14/2013 11:20	**Dilution:**	1	**File ID:**	I2_051613-10
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.89		0.200	0.100
Nitrate	14797-55-8	0.543	J	0.600	0.100
Sulfate	14808-79-8	37.0		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L13050733
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050733-09
Client ID: 54MW05
Matrix: Water
Workgroup #: WG430447
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/16/2013 11:25
Cal Date: 04/10/2013 13:38
Run Date: 05/16/2013 14:39
File ID: I2_051613-11

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.51		0.200	0.100
Nitrate	14797-55-8	1.60		0.600	0.100
Sulfate	14808-79-8	15.7		1.00	0.500

Sample #: L13050733-11
Client ID: 54TMW02
Matrix: Water
Workgroup #: WG430447
Collect Date: 05/14/2013 12:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/16/2013 11:25
Cal Date: 04/10/2013 13:38
Run Date: 05/16/2013 14:57
File ID: I2_051613-12

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.57		0.200	0.100
Nitrate	14797-55-8	1.60		0.600	0.100
Sulfate	14808-79-8	15.6		1.00	0.500

Sample #: L13050733-13
Client ID: 54MW01
Matrix: Water
Workgroup #: WG430447
Collect Date: 05/14/2013 13:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC2
Prep Date: 05/16/2013 11:25
Cal Date: 04/10/2013 13:38
Run Date: 05/16/2013 15:16
File ID: I2_051613-13

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	2.11		0.200	0.100
Nitrate	14797-55-8	0.368	J J	0.600	0.100
Sulfate	14808-79-8	26.1		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L13020681

DATE: June 21, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 21, 2013. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of fourteen aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW13	L13020681-01	54MW12	L13020681-05
54MW13	L13020681-02	54MW12	L13020681-06
54MW02	L13020681-03	54RB22113	L13020681-07
54MW02	L13020681-04	54RB22113	L13020681-08

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/21/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13020681

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/21/13, the cooler was received on 02/22/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C. Even though the receipt temperature was below criteria for the cooler; there were no impacts to the data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The samples were collected on 02/21/13. The TOC and TIC analysis were run on 02/23/13. The DOC and DIC analysis were run on 02/25/13. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC:	1 - blank
	5 - standards ($r \geq 0.995$)
	ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 02/23/13. The DOC and DIC analysis were run on 02/25/13. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13020681-01), 54MW13 (L13020681-02), 54MW02 (L13020681-03), 54MW02 (L13020681-04), 54MW12 (L13020681-05), 54MW12 (L13020681-06), 54RB22113 (L13020681-07), and 54RB22113 (L13020681-08) apply to these initial and continuing calibrations.

III-Blank Analysis
The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-08) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB22113 (L13020681-07) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
02/23/13	TOC	ICB/CCBs	<LOD	NA	None
02/22/13	TOC	WG421588-01	<½MRL	NA	None
02/25/13	DOC	WG421590-01	<½MRL	NA	None
02/23/13	TOC	54RB22113	0.653J	3.27	54MW13, 54MW02, 54MW12
02/23/13	TIC	54RB22113	<½MRL	NA	None
02/25/13	DOC	54RB22113	0.950J	4.75	54MW13, 54MW02, 54MW12
02/25/13	DIC	54RB22113	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG421588-02 and WG421588-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 02/22/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13020681-01), 54MW02 (L13020681-03), 54MW12 (L13020681-05), and 54RB22113 (L13020681-07) apply to this LCS/LCSD.

- Samples WG421590-02 and WG421590-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 02/25/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13020681-02), 54MW02 (L13020681-04), 54MW12 (L13020681-06), and 54RB22113 (L13020681-08) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- No site specific aqueous MS/MSD was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG; therefore, was not evaluated. See LCS/LCSD Section IV for laboratory precision and accuracy statements.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG; therefore, was not evaluated.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW13 (L13020681-01), 54MW13 (L13020681-02), 54MW02 (L13020681-03), 54MW02 (L13020681-04), 54MW12 (L13020681-05), 54MW12 (L13020681-06), 54RB22113 (L13020681-07), and 54RB22113 (L13020681-08) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW13	TOC/DOC	1.0; 2.0	0.683J	2.08	67.2	J
54MW13	TIC/DIC	1.0; 2.0	2.75	44.2	93.8	J
54MW02	TOC/DOC	1.0; 2.0	0.916J	2.90	68.4	J
54MW02	TIC/DIC	1.0; 2.0	4.25	80.4	94.7	J
54MW12	TOC/DOC	1.0; 2.0	1.21	2.49	51.4	J
54MW12	TIC/DIC	1.0; 2.0	4.15	55.3	92.5	J
54RB22113	TOC/DOC	1.0; 2.0	0.653J	0.950J	31.3	J
54RB22113	TIC/DIC	1.0; 2.0	<1.00	<1.00	NA	None

J = Estimated value.

Sample: 54MW13 (L13020681-01), TOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 133.9
DF = 1

X = (3.43 mg/L) * 1 = 3.43 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 86.78
DF = 1

X = (2.75 mg/L) * 1 = 2.75 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 3.43 – 2.75 = 0.684 mg/L

Reported Value = 0.683 mg/L
% Difference = 0.2%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW13	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/23/2013 04:21
Collect Date:	02/21/2013 08:00	**Dilution:**	1	**File ID:**	TC02222013.066
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.683	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Microbac

Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020681-01
Client ID: 54MW13
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/21/2013 08:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/23/2013 04:21
File ID: TC02222013.066

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		2.75	J	1.00	0.500

Sample #: L13020681-02
Client ID: 54MW13
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/21/2013 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 17:03
File ID: TC2252013..024

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.08	B	2.00	1.00

Sample #: L13020681-02
Client ID: 54MW13
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/21/2013 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 17:03
File ID: TC2252013..024

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		44.2	J	2.00	1.00

Sample #: L13020681-03
Client ID: 54MW02
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/21/2013 09:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/23/2013 04:33
File ID: TC02222013.067

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		4.25	J	1.00	0.500

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Microbac

Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-03	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW02	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/23/2013 04:33
Collect Date:	02/21/2013 09:20	**Dilution:**	1	**File ID:**	TC02222013.067
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.916	J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Certificate of Analysis

Sample #: L13020681-04 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW02 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421590 **Analyst:** BAF **Run Date:** 02/25/2013 17:17
Collect Date: 02/21/2013 09:20 **Dilution:** 2 **File ID:** TC2252013..025
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		80.4	J	2.00	1.00

Sample #: L13020681-04 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW02 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421590 **Analyst:** BAF **Run Date:** 02/25/2013 17:17
Collect Date: 02/21/2013 09:20 **Dilution:** 2 **File ID:** TC2252013..025
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.90	B	2.00	1.00

Sample #: L13020681-05 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW12 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421588 **Analyst:** BAF **Run Date:** 02/23/2013 04:46
Collect Date: 02/21/2013 10:40 **Dilution:** 1 **File ID:** TC02222013.068
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		4.15	J	1.00	0.500

Sample #: L13020681-05 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW12 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421588 **Analyst:** BAF **Run Date:** 02/23/2013 04:46
Collect Date: 02/21/2013 10:40 **Dilution:** 1 **File ID:** TC02222013.068
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.21	B	1.00	0.500

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Microbac

Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020681-06
Client ID: 54MW12
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/21/2013 10:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 17:52
File ID: TC2252013..028

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		55.3	J	2.00	1.00

Sample #: L13020681-06
Client ID: 54MW12
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/21/2013 10:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 17:52
File ID: TC2252013..028

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.49	B	2.00	1.00

Sample #: L13020681-07
Client ID: 54RB22113
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/21/2013 12:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/23/2013 04:58
File ID: TC02222013.069

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.653	J J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB22113	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/23/2013 04:58
Collect Date:	02/21/2013 12:00	**Dilution:**	1	**File ID:**	TC02222013.069
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-08	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB22113	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 18:05
Collect Date:	02/21/2013 12:00	**Dilution:**	1	**File ID:**	TC2252013..029
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	0.950	J J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-08	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB22113	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 18:05
Collect Date:	02/21/2013 12:00	**Dilution:**	1	**File ID:**	TC2252013..029
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13020681

DATE: June 20, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 21, 2013. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of four aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW13	L13020681-01	54MW12	L13020681-05
54MW02	L13020681-03	54RB22113	L13020681-07

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable.

Eric Malarek, Chemist

6/20/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13020681

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/21/13, the cooler was received on 02/22/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C. Even though the receipt temperature was below criteria for the cooler; there were no impacts to the data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 02/21/13 for perchlorate analysis. The aqueous samples were prepped and analyzed on 03/01/13. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check

LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:
1- blank (<1/2MRL DoD Perchlorate Handbook)
5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
ICV (≤15%D DoD Perchlorate Handbook)
CCV/ICS (≤15%D DoD Perchlorate Handbook)
LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 01/29/13 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW13 (L13020681-01), 54MW02 (L13020681-03), 54MW12 (L13020681-05), and 54RB22113 (L13020681-07) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
03/01/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
03/01/13	WG422283-02	All perchlorate <½MRL	NA	NA	None
03/01/13	54RB22113	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG422283-03 was used as aqueous LCS for perchlorate analysis dated 03/01/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13020681-01), 54MW02 (L13020681-03), 54MW12 (L13020681-05), and 54RB22113 (L13020681-07) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- No site specific aqueous MS/MSD was analyzed for perchlorate analysis in this SDG; therefore, was not evaluated. See LCS Section VII for laboratory accuracy statement.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for perchlorate analysis in this SDG; therefore, was not evaluated.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW13 (L13020681-01), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.19
b = 0.000928
Y = Area = 22300/296000 = 0.07533
X = 0.06253

Conc. μg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in μg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (μg/L)
DF = Dilution factor

Conc. μg/L = (0.06253 * 5 * 1) = 0.313 μg/L (Signal #1)
Reported Value = 0.313 μg/L
% Difference = 0.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020681-01
Client ID: 54MW13
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/21/2013 08:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 16:05
File ID: 1LM.LM19925

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.313		0.200	0.100

Sample #: L13020681-03
Client ID: 54MW02
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/21/2013 09:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 16:24
File ID: 1LM.LM19926

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	1.41		0.200	0.100

Sample #: L13020681-05
Client ID: 54MW12
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/21/2013 10:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 16:43
File ID: 1LM.LM19927

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	2.98		0.200	0.100

Sample #: L13020681-07
Client ID: 54RB22113
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/21/2013 12:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 17:21
File ID: 1LM.LM19929

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13020681

DATE: June 20, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 21, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of eight aqueous samples (includes four re-analysis samples) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW13	L13020681-01	54MW12	L13020681-05
54MW13	L13020681-01RE	54MW12	L13020681-05RE
54MW02	L13020681-03	54RB22113	L13020681-07
54MW02	L13020681-03RE	54RB22113	L13020681-07RE

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
X		Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/20/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13020681

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/21/13, the cooler was received on 02/22/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C. Even though the receipt temperature was below criteria for the cooler; there were no impacts to the data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 02/21/13. The samples were extracted on 02/27/13 and were analyzed on 02/27/13 and 02/28/13 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
02/27/13	WG422024-01	All target explosives <½MRL	NA	NA	None
02/28/13	WG422024-01	All target explosives <½MRL	NA	NA	None
02/28/13	54RB22113	2,4,6-Trinitrotoluene	1.05J	5.25	54MW13

J = Estimated Value <MRL and >MDL.
NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW13 (L13020681-01), 54MW02 (L13020681-03), 54MW12 (L13020681-05), and 54RB22113 (L13020681-07) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. No confirmation samples were required for all associated samples were non-detect. Samples 54MW10 54MW13 (L13020681-01RE), 54MW02 (L13020681-03RE), 54MW12 (L13020681-05RE), and 54RB22113 (L13020681-07RE) apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 02/27/13 @12:05 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 02/27/13 @21:58 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW13 (L13020681-01), 54MW02 (L13020681-03), 54MW12 (L13020681-05), and 54RB22113 (L13020681-07) were reported using this continuing calibration.

- For explosives continuing calibration performed on 02/28/13 @06:44 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 02/15/12 @20:05 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No aqueous confirmation samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 02/28/13 @11:53 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation samples 54MW10 54MW13 (L13020681-01RE), 54MW02 (L13020681-03RE), 54MW12 (L13020681-05RE), and 54RB22113 (L13020681-07RE) apply to this continuing calibration.

- For explosives continuing calibration performed on 02/28/13 @21:56 on instrument HPLC4, all criteria were met. No qualifiers were applied. No aqueous confirmation samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample
Laboratory control samples (LCS) are used to monitor long-term accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used.

- Sample WG422024-02 was used as the aqueous LCS for the explosives analysis on 02/27/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13020681-01), 54MW02 (L13020681-03), 54MW12 (L13020681-05), and 54RB22113 (L13020681-07) apply to this LCS.

- Sample WG422024-02 was used as the aqueous LCS for the explosives analysis on 02/28/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13020681-01RE), 54MW02 (L13020681-03RE), 54MW12 (L13020681-05RE), and 54RB22113 (L13020681-07RE) apply to this LCS.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- No site specific aqueous MS/MSD was analyzed for explosives analysis in this SDG; therefore, was not evaluated. See LCS/LCSD Section VI for laboratory precision and accuracy statements.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for explosives analysis in this SDG; therefore, was not evaluated.

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- For sample 54RB22113 (L13020681-07), target compound 2,4,6-trinitrotoluene (66.2%) was outside confirmation criteria; therefore, were qualified estimated based upon the high RPD.

- All other positive detections met confirmation criteria for all detected target analytes in this SDG.

Sample: 54MW02 (L13020681-03), 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.26899837
b = -0.1564446
Y = Area = 24.78124

X = 92.71

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (92.71 * 10 * 1) / (920) = 1.01 µg/L (Signal #1)

Reported Value = 1.01 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW13	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/27/2013 23:16
Collect Date:	02/21/2013 08:00	**Dilution:**	1	**File ID:**	5L009193.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.06	0.266
2,4-Dinitrotoluene	121-14-2		U	1.06	0.266
2,6-Dinitrotoluene	606-20-2		U	1.06	0.266
2,4,6-Trinitrotoluene	118-96-7	0.318	J B	1.06	0.266
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.06	0.266
2-Nitrotoluene	88-72-2		U	1.06	0.266
4-Nitrotoluene	99-99-0		U	1.06	0.266
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.06	0.266
RDX	121-82-4	0.642	J J	1.06	0.266
Nitroglycerin	55-63-0		U	1.06	0.266

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	96.8	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW13	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/28/2013 16:29
Collect Date:	02/21/2013 08:00	**Dilution:**	1	**File ID:**	4L024836.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.06	0.266
2,4-Dinitrotoluene	121-14-2		U	1.06	0.266
2,6-Dinitrotoluene	606-20-2		U	1.06	0.266
2,4,6-Trinitrotoluene	118-96-7	0.394	J B	1.06	0.266
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.06	0.266
2-Nitrotoluene	88-72-2		U	1.06	0.266
4-Nitrotoluene	99-99-0		U	1.06	0.266
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.06	0.266
RDX	121-82-4	0.496	J J	1.06	0.266
Nitroglycerin	55-63-0		U	1.06	0.266

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	96.1	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020681-03
Client ID: 54MW02
Matrix: Water
Workgroup #: WG422133
Collect Date: 02/21/2013 09:20
Sample Tag: CF01

PrePrep Method: N/A
Prep Method: METHOD
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC4
Prep Date: 02/27/2013 08:21
Cal Date: 02/15/2012 19:12
Run Date: 02/28/2013 17:22
File ID: 4L024837.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.09	0.272
2,4-Dinitrotoluene	121-14-2		U	1.09	0.272
2,6-Dinitrotoluene	606-20-2		U	1.09	0.272
2,4,6-Trinitrotoluene	118-96-7		U	1.09	0.272
2-Amino-4,6-dinitrotoluene	35572-78-2	0.970	J J	1.09	0.272
2-Nitrotoluene	88-72-2		U	1.09	0.272
4-Nitrotoluene	99-99-0		U	1.09	0.272
4-Amino-2,6-dinitrotoluene	19406-51-0	1.02	J J	1.09	0.272
RDX	121-82-4		U	1.09	0.272
Nitroglycerin	55-63-0		U	1.09	0.272

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	103	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW02	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/27/2013 23:55
Collect Date:	02/21/2013 09:20	**Dilution:**	1	**File ID:**	5L009194.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.09	0.272
2,4-Dinitrotoluene	121-14-2		U	1.09	0.272
2,6-Dinitrotoluene	606-20-2		U	1.09	0.272
2,4,6-Trinitrotoluene	118-96-7		U	1.09	0.272
2-Amino-4,6-dinitrotoluene	35572-78-2	1.09	J	1.09	0.272
2-Nitrotoluene	88-72-2		U	1.09	0.272
4-Nitrotoluene	99-99-0		U	1.09	0.272
4-Amino-2,6-dinitrotoluene	19406-51-0	1.01	J	1.09	0.272
RDX	121-82-4		U	1.09	0.272
Nitroglycerin	55-63-0		U	1.09	0.272

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	104	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW12	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/28/2013 00:34
Collect Date:	02/21/2013 10:40	**Dilution:**	1	**File ID:**	5L009195.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7	6.29		1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2	2.95		1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0	1.82		1.10	0.275
RDX	121-82-4	3.79		1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	95.9	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW12	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/28/2013 18:15
Collect Date:	02/21/2013 10:40	**Dilution:**	1	**File ID:**	4L024838.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7	6.11		1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2	2.84		1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0	1.64		1.10	0.275
RDX	121-82-4	2.73		1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	98.8	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020681-07
Client ID: 54RB22113
Matrix: Water
Workgroup #: WG422133
Collect Date: 02/21/2013 12:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: METHOD
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 02/27/2013 08:21
Cal Date: 02/23/2012 15:43
Run Date: 02/28/2013 01:13
File ID: 5L009196.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.09	0.272
2,4-Dinitrotoluene	121-14-2		U	1.09	0.272
2,6-Dinitrotoluene	606-20-2		U	1.09	0.272
2,4,6-Trinitrotoluene	118-96-7	1.05	J	1.09	0.272
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.09	0.272
2-Nitrotoluene	88-72-2		U	1.09	0.272
4-Nitrotoluene	99-99-0		U	1.09	0.272
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.09	0.272
RDX	121-82-4		U	1.09	0.272
Nitroglycerin	55-63-0		U	1.09	0.272

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	105	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-07	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54RB22113	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/28/2013 19:08
Collect Date:	02/21/2013 12:00	**Dilution:**	1	**File ID:**	4L024839.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.09	0.272
2,4-Dinitrotoluene	121-14-2		U	1.09	0.272
2,6-Dinitrotoluene	606-20-2		U	1.09	0.272
2,4,6-Trinitrotoluene	118-96-7	0.528	J J	1.09	0.272
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.09	0.272
2-Nitrotoluene	88-72-2		U	1.09	0.272
4-Nitrotoluene	99-99-0		U	1.09	0.272
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.09	0.272
RDX	121-82-4		U	1.09	0.272
Nitroglycerin	55-63-0		U	1.09	0.272

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	103	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13020681

DATE: June 20, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 21, 2013. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of four aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW13	L13020681-01	54MW12	L13020681-05
54MW02	L13020681-03	54RB22113	L13020681-07

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

.

Eric Malarek, Chemist

6/20/13

Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13020681

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/21/13, the cooler was received on 02/22/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C. Even though the receipt temperature was below criteria for the cooler; there were no impacts to the data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: Samples were collected on 02/21/13. The samples were prepped and analyzed on 02/22/13 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 11/29/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW13 (L13020681-01), 54MW02 (L13020681-03), 54MW12 (L13020681-05), and 54RB22113 (L13020681-07) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
02/22/13	Chloride	ICB/CCBs	<LOD	NA	None
02/22/13	Sulfate	ICB/CCBs	<LOD	NA	None
02/22/13	Nitrate	ICB/CCBs	<LOD	NA	None
02/22/13	Chloride	WG421685-01	<½MRL	NA	None
02/22/13	Sulfate	WG421685-01	<½MRL	NA	None
02/22/13	Nitrate	WG421685-01	<½MRL	NA	None
02/22/13	Chloride	54RB22113	<½MRL	NA	None
02/22/13	Sulfate	54RB22113	<½MRL	NA	None
02/22/13	Nitrate	54RB22113	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG421685-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 02/22/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13020681-01), 54MW02 (L13020681-03), 54MW12 (L13020681-05), and 54RB22113 (L13020681-07) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate

Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at

a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- No site specific aqueous MS/MSD was analyzed for chloride, sulfate, and nitrate analysis in this SDG; therefore, was not evaluated. See LCS Section IV for laboratory accuracy statement.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for chloride, sulfate, and nitrate analysis in this SDG; therefore, was not evaluated.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW13 (L13020681-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1331
b = -0.0222
Y = Area = 3.052
DF = 1

X = 23.10 mg/L * DF = 23.1 mg/L * 1 = 23.1 mg/L

Reported concentration = 23.1 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-01	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW13	**Prep Method:**	9056	**Prep Date:**	02/22/2013 15:30
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421685	**Analyst:**	JBK	**Run Date:**	02/22/2013 20:56
Collect Date:	02/21/2013 08:00	**Dilution:**	1	**File ID:**	I1_022213-22
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.92		0.200	0.100
Nitrate	14797-55-8	0.501	J J	0.600	0.100
Sulfate	14808-79-8	23.1		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020681-03	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW02	**Prep Method:**	9056	**Prep Date:**	02/22/2013 15:30
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421685	**Analyst:**	JBK	**Run Date:**	02/22/2013 21:14
Collect Date:	02/21/2013 09:20	**Dilution:**	1	**File ID:**	I1_022213-23
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.30		0.200	0.100
Nitrate	14797-55-8	0.315	J J	0.600	0.100
Sulfate	14808-79-8	36.8		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L13020681
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020681-05
Client ID: 54MW12
Matrix: Water
Workgroup #: WG421685
Collect Date: 02/21/2013 10:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 02/22/2013 15:30
Cal Date: 11/29/2012 10:40
Run Date: 02/22/2013 21:31
File ID: I1_022213-24

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.85		0.200	0.100
Nitrate	14797-55-8	1.95		0.600	0.100
Sulfate	14808-79-8	35.8		1.00	0.500

Sample #: L13020681-07
Client ID: 54RB22113
Matrix: Water
Workgroup #: WG421685
Collect Date: 02/21/2013 12:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 02/22/2013 15:30
Cal Date: 11/29/2012 10:40
Run Date: 02/22/2013 21:49
File ID: I1_022213-25

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6		U	0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8		U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L13020654

DATE: June 19, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 20, 2013. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of fourteen aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L13020654-01	54MW08	L13020654-08
54MW09	L13020654-02	54MW10	L13020654-13
54TM09	L13020654-03	54MW10	L13020654-14
54TM09	L13020654-04	54MW05	L13020654-15
54MW11	L13020654-05	54MW05	L13020654-16
54MW11	L13020654-06	54MW03	L13020654-17
54MW08	L13020654-07	54MW03	L13020654-18

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
X		Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/19/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13020654

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/20/13, the coolers were received on 02/21/13 by the primary laboratory (Microbac Laboratories, Inc.) at 2.0°C, 0.0°C, and 1.0°C. Even though the receipt temperatures were below criteria for the coolers; there were no impacts to the data quality and no qualifiers were applied based upon these outliers.

- Holding Time Review: The samples were collected on 02/20/13. The TOC and TIC analysis were run on 02/22/13. The DOC and DIC analysis were run on 02/25/13. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC:	1 - blank
	5 - standards ($r \geq 0.995$)
	ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 02/22/13. The DOC and DIC analysis were run on 02/25/13. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54MW09 (L13020654-02), 54TM09 (L13020654-03), 54TM09 (L13020654-04), 54MW11 (L13020654-05), 54MW11 (L13020654-06), 54MW08 (L13020654-07), 54MW08 (L13020654-08), 54MW10 (L13020654-13), 54MW10 (L13020654-14), 54MW05 (L13020654-15), 54MW05 (L13020654-16), 54MW03 (L13020654-17), and 54MW03 (L13020654-18) apply to these initial and continuing calibrations.

III-Blank Analysis
The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-08) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB22113 (L13020681-07) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
02/22/13	TOC	ICB/CCBs	<LOD	NA	None
02/22/13	TOC	WG421588-01	<½MRL	NA	None
02/25/13	DOC	WG421590-01	<½MRL	NA	None
02/23/13	TOC	54RB22113	0.653J	3.27	54MW09, 54TM09, 54MW11, 54MW10, 54MW03
02/23/13	TIC	54RB22113	<½MRL	NA	None
02/25/13	DOC	54RB22113	0.950J	4.75	54MW09, 54TM09, 54MW11, 54MW08, 54MW10, 54MW03
02/25/13	DIC	54RB22113	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG421588-02 and WG421588-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 02/22/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this LCS/LCSD.

- Samples WG421590-02 and WG421590-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 02/25/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-02), 54TM09 (L13020654-04), 54MW11 (L13020654-06), 54MW08 (L13020654-08), 54MW10 (L13020654-14), 54MW05 (L13020654-16), and 54MW03 (L13020654-18) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- Sample 54MW08 (L13020654-07) was used as the aqueous MS/MSD for TOC and TIC analysis on 02/22/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this MS/MSD.

- Sample 54MW08 (L13020654-08) was used as the aqueous MS/MSD for DOC and DIC analysis on 02/25/13. DOC (116%) was outside criteria. All criteria were met for DIC. For DOC, all associated samples were qualified estimated bias high "K" for detections and no qualification for non-detections based upon the high recovery. Samples 54MW09 (L13020654-02), 54TM09 (L13020654-04), 54MW11 (L13020654-06), 54MW08 (L13020654-08), 54MW10 (L13020654-14), 54MW05 (L13020654-16), and 54MW03 (L13020654-18) apply to this MS/MSD.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW09 (L13020654-01) and 54TM09 (L13020654-03) was collected for TOC and TIC analysis. TOC was detected at 2.09 mg/L in the parent sample and at 2.34 mg/L in the duplicate pair resulting in a RPD of 11.3% (within limits). TIC was detected at 12.4 mg/L in the parent sample and at 12.1 mg/L in the duplicate pair resulting in a RPD of 2.5% (within limits). All criteria were met. No qualifiers were applied.

- Field aqueous sample duplicate pair 54MW09 (L13020654-02) and 54TM09 (L13020654-04) was collected for DOC and DIC analysis. DOC was detected at 2.39 mg/L in the parent sample and at 2.97 mg/L in the duplicate pair resulting in a RPD of 21.6% (within limits). DIC was detected at 35.3 mg/L in the parent sample and at 35.5 mg/L in the duplicate pair resulting in a RPD of 0.6% (within limits). All criteria were met for DIC. All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW09 (L13020654-01), 54MW09 (L13020654-02), 54TM09 (L13020654-03), 54TM09 (L13020654-04), 54MW11 (L13020654-05), 54MW11 (L13020654-06), 54MW08 (L13020654-07), 54MW08 (L13020654-08), 54MW10 (L13020654-13), 54MW10 (L13020654-14), 54MW05 (L13020654-15), 54MW05 (L13020654-16), 54MW03 (L13020654-17), and 54MW03 (L13020654-18) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW09	TOC/DOC	1.0; 2.0	2.09	2.39	12.6	J
54MW09	TIC/DIC	1.0; 2.0	12.4	35.3	64.9	J
54TM09	TOC/DOC	1.0; 2.0	2.34	2.97	21.2	J
54TM09	TIC/DIC	1.0; 2.0	12.1	35.5	65.9	J
54MW11	TOC/DOC	1.0; 2.0	1.88	1.24	51.6	J
54MW11	TIC/DIC	1.0; 2.0	19.2	58.4	67.1	J
54MW08	TOC/DOC	1.0; 2.0	<1.00	1.42J	NA	None
54MW08	TIC/DIC	1.0; 2.0	23.0	55.8	58.8	J
54MW10	TOC/DOC	1.0; 2.0	1.14	1.47J	22.5	J
54MW10	TIC/DIC	1.0; 2.0	24.7	56.9	56.7	J
54MW05	TOC/DOC	1.0; 2.0	<1.00	<2.00	0.0	None
54MW05	TIC/DIC	1.0; 2.0	22.7	54.2	58.1	J
54MW03	TOC/DOC	1.0; 2.0	1.07	2.32	53.9	J
54MW03	TIC/DIC	1.0; 2.0	25.5	64.5	60.5	J

Sample: 54MW09 (L13020654-01), TOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 558.5
DF = 1

X = (14.47 mg/L) * 1 = 14.47 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 367.8
DF = 1

X = (12.38 mg/L) * 1 = 12.38 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 14.47 – 12.38 = 2.09 mg/L

Reported Value = 2.09 mg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-01
Client ID: 54MW09
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/20/2013 08:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 15:25
File ID: TC02222013.012

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.09	B	1.00	0.500

Sample #: L13020654-01
Client ID: 54MW09
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/20/2013 08:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 15:25
File ID: TC02222013.012

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		12.4	J	1.00	0.500

Sample #: L13020654-02
Client ID: 54MW09
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/20/2013 08:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 14:26
File ID: TC2252013..012

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		35.3	J	2.00	1.00

Sample #: L13020654-02
Client ID: 54MW09
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/20/2013 08:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 14:26
File ID: TC2252013..012

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.39	B	2.00	1.00

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-03
Client ID: 54TM09
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/20/2013 08:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 15:38
File ID: TC02222013.013

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.34	B	1.00	0.500

Sample #: L13020654-03
Client ID: 54TM09
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/20/2013 08:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 15:38
File ID: TC02222013.013

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		12.1	J	1.00	0.500

Sample #: L13020654-04
Client ID: 54TM09
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/20/2013 08:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 14:40
File ID: TC2252013..013

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.97	B	2.00	1.00

Sample #: L13020654-04
Client ID: 54TM09
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/20/2013 08:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 14:40
File ID: TC2252013..013

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		35.5	J	2.00	1.00

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Certificate of Analysis

Sample #: L13020654-05 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW11 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421588 **Analyst:** BAF **Run Date:** 02/22/2013 16:13
Collect Date: 02/20/2013 10:00 **Dilution:** 1 **File ID:** TC02222013.016
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		19.2	J	1.00	0.500

Sample #: L13020654-05 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW11 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421588 **Analyst:** BAF **Run Date:** 02/22/2013 16:13
Collect Date: 02/20/2013 10:00 **Dilution:** 1 **File ID:** TC02222013.016
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.88	B	1.00	0.500

Sample #: L13020654-06 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW11 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421590 **Analyst:** BAF **Run Date:** 02/25/2013 15:15
Collect Date: 02/20/2013 10:00 **Dilution:** 2 **File ID:** TC2252013..016
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		58.4	J	2.00	1.00

Sample #: L13020654-06 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW11 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421590 **Analyst:** BAF **Run Date:** 02/25/2013 15:15
Collect Date: 02/20/2013 10:00 **Dilution:** 2 **File ID:** TC2252013..016
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.24	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW08	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 16:26
Collect Date:	02/20/2013 11:05	**Dilution:**	1	**File ID:**	TC02222013.017
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC		U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-07
Client ID: 54MW08
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/20/2013 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 16:26
File ID: TC02222013.017

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		23.0	J	1.00	0.500

Sample #: L13020654-08
Client ID: 54MW08
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/20/2013 11:05
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 15:28
File ID: TC2252013..017

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		55.8	J	2.00	1.00

Sample #: L13020654-08
Client ID: 54MW08
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/20/2013 11:05
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 15:28
File ID: TC2252013..017

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.42	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-13	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 17:19
Collect Date:	02/20/2013 12:35	**Dilution:**	1	**File ID:**	TC02222013.021
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.14	B	1.00	0.500

Sample #:	L13020654-13	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 17:19
Collect Date:	02/20/2013 12:35	**Dilution:**	1	**File ID:**	TC02222013.021
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		24.7	J	1.00	0.500

Sample #:	L13020654-14	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 16:22
Collect Date:	02/20/2013 12:35	**Dilution:**	2	**File ID:**	TC2252013..021
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.47	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Sample #: L13020654-14 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW10 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421590 **Analyst:** BAF **Run Date:** 02/25/2013 16:22
Collect Date: 02/20/2013 12:35 **Dilution:** 2 **File ID:** TC2252013..021
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		56.9	J	2.00	1.00

Sample #: L13020654-15 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW05 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421588 **Analyst:** BAF **Run Date:** 02/22/2013 17:32
Collect Date: 02/20/2013 13:35 **Dilution:** 1 **File ID:** TC02222013.022
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		22.7	J	1.00	0.500

Sample #: L13020654-15 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW05 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG421588 **Analyst:** BAF **Run Date:** 02/22/2013 17:32
Collect Date: 02/20/2013 13:35 **Dilution:** 1 **File ID:** TC02222013.022
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC		U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-16	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW05	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 16:36
Collect Date:	02/20/2013 13:35	**Dilution:**	2	**File ID:**	TC2252013..022
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC		U	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-16
Client ID: 54MW05
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/20/2013 13:35
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 16:36
File ID: TC2252013..022

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		54.2	J	2.00	1.00

Sample #: L13020654-17
Client ID: 54MW03
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/20/2013 14:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 17:45
File ID: TC02222013.023

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		25.5	J	1.00	0.500

Sample #: L13020654-17
Client ID: 54MW03
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/20/2013 14:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 17:45
File ID: TC02222013.023

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.07	B	1.00	0.500

Sample #: L13020654-18
Client ID: 54MW03
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/20/2013 14:25
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 16:49
File ID: TC2252013..023

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		64.5	J	2.00	1.00

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Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-18	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW03	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 16:49
Collect Date:	02/20/2013 14:25	**Dilution:**	2	**File ID:**	TC2252013..023
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.32	B	2.00	1.00

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13020654

DATE: June 18, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 20, 2013. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of seven aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L13020654-01	54MW10	L13020654-13
54TM09	L13020654-03	54MW05	L13020654-15
54MW11	L13020654-05	54MW03	L13020654-17
54MW08	L13020654-07		

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13020654

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/20/13, the coolers were received on 02/21/13 by the primary laboratory (Microbac Laboratories, Inc.) at 2.0°C, 0.0°C, and 1.0°C. Even though the receipt temperatures were below criteria for the coolers; there were no impacts to the data quality and no qualifiers were applied based upon these outliers.

- Holding Time Review: The aqueous samples were collected on 02/20/13 for perchlorate analysis. The aqueous samples were prepped and analyzed on 03/01/13. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check
LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:
1- blank (<1/2MRL DoD Perchlorate Handbook)
5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
ICV (≤15%D DoD Perchlorate Handbook)
CCV/ICS (≤15%D DoD Perchlorate Handbook)
LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 01/29/13 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
03/01/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
03/01/13	WG422283-02	All perchlorate <½MRL	NA	NA	None
03/01/13	54RB22113	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG422283-03 was used as aqueous LCS for perchlorate analysis dated 03/01/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- Sample 54MW08 (L13020654-07) was used as the aqueous MS/MSD for the perchlorate analysis on 03/01/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this MS/MSD.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW09 (L13020654-01) and 54TM09 (L13020654-03) was analyzed for perchlorate analysis in this SDG. Perchlorate was detected in the original sample at 1.07 µg/L and in the duplicate pair at 1.03 µg/L; resulting in a RPD of 3.8%. All criteria were met. No qualifiers were applied.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW09 (L13020654-01), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.19
b = 0.000928
Y = Area = 92100/359000 = 0.2565
X = 0.2148

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.2148 * 5 * 1) = 1.07 µg/L (Signal #1)
Reported Value = 1.07 µg/L
% Difference = 0.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-01
Client ID: 54MW09
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/20/2013 08:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 11:40
File ID: 1LM.LM19911

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	1.07		0.200	0.100

Sample #: L13020654-03
Client ID: 54TM09
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/20/2013 08:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 11:59
File ID: 1LM.LM19912

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	1.03		0.200	0.100

Sample #: L13020654-05
Client ID: 54MW11
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/20/2013 10:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 12:18
File ID: 1LM.LM19913

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.263	J	0.200	0.100

Sample #: L13020654-07
Client ID: 54MW08
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/20/2013 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 12:36
File ID: 1LM.LM19914

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.484		0.200	0.100

Form I Copy

Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-09
Client ID: 54MW08-MS
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/20/2013 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 12:55
File ID: 1LM.LM19915

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.661		0.200	0.100

Sample #: L13020654-11
Client ID: 54MW08-MSD
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/20/2013 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 13:14
File ID: 1LM.LM19916

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.667		0.200	0.100

Sample #: L13020654-13
Client ID: 54MW10
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/20/2013 12:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 13:33
File ID: 1LM.LM19917

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.832		0.200	0.100

Sample #: L13020654-15
Client ID: 54MW05
Matrix: Water
Workgroup #: WG422283
Collect Date: 02/20/2013 13:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 03/01/2013 09:30
Cal Date: 01/29/2013 19:25
Run Date: 03/01/2013 15:08
File ID: 1LM.LM19922

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.255	J	0.200	0.100

Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-17	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW03	**Prep Method:**	6850	**Prep Date:**	03/01/2013 09:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	01/29/2013 19:25
Workgroup #:	WG422283	**Analyst:**	JWR	**Run Date:**	03/01/2013 15:27
Collect Date:	02/20/2013 14:25	**Dilution:**	1	**File ID:**	1LM.LM19923
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.309		0.200	0.100

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13020654

DATE: June 18, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 20, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of nine aqueous samples (includes two re-analysis samples) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L13020654-01	54MW10	L13020654-13RE
54TM09	L13020654-03	54MW05	L13020654-15
54MW11	L13020654-05	54MW03	L13020654-17
54MW08	L13020654-07	54MW03	L13020654-17RE
54MW10	L13020654-13		

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
X		Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/18/13
Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13020654

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/20/13, the coolers were received on 02/21/13 by the primary laboratory (Microbac Laboratories, Inc.) at 2.0°C, 0.0°C, and 1.0°C. Even though the receipt temperatures were below criteria for the coolers; there were no impacts to the data quality and no qualifiers were applied based upon these outliers.

- Holding Time Review: The aqueous samples were collected on 02/20/13. The samples were extracted on 02/27/13 and were analyzed on 02/27/13 and 02/28/13 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
02/27/13	WG422024-01	All target explosives <½MRL	NA	NA	None
02/28/13	WG422024-01	All target explosives <½MRL	NA	NA	None
02/28/13	54RB22113	2,4,6-Trinitrotoluene	1.05J	5.25	54MW10

J = Estimated Value <MRL and >MDL.
NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. No confirmation samples were required for all associated samples were non-detect. Samples 54MW10 (L13020654-13RE) and 54MW03 (L13020654-17RE) apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 02/27/13 @12:05 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), and 54MW05 (L13020654-15) were reported using this continuing calibration.

- For explosives continuing calibration performed on 02/27/13 @21:58 on instrument HPLC5, all criteria were met. No qualifiers were applied. Sample 54MW03 (L13020654-17) was reported using this continuing calibration.

- For explosives continuing calibration performed on 02/28/13 @06:44 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 02/15/12 @20:05 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No aqueous confirmation samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 02/28/13 @11:53 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation samples 54MW10 (L13020654-13RE) and 54MW03 (L13020654-17RE) apply to this continuing calibration.

- For explosives continuing calibration performed on 02/28/13 @21:56 on instrument HPLC4, all criteria were met. No qualifiers were applied. No aqueous confirmation samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample
Laboratory control samples (LCS) are used to monitor long-term accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used.

- Sample WG422024-02 was used as the aqueous LCS for the explosives analysis on 02/27/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this LCS.

- Sample WG422024-02 was used as the aqueous LCS for the explosives analysis on 02/28/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (L13020654-13RE) and 54MW03 (L13020654-17RE) apply to this LCS.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Sample 54MW08 (L13020654-07) was used as the aqueous MS/MSD for the explosives analysis on 02/27/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW10 (L13020654-13RE), 54MW05 (L13020654-15), 54MW03 (L13020654-17), and 54MW03 (L13020654-17RE) apply to this MS/MSD.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW09 (L13020654-01) and 54TM09 (L13020654-03) was analyzed for explosives analysis in this SDG. All target explosives were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- All positive detections met confirmation criteria for all detected target analytes in this SDG. No qualifiers were applied.

Sample: 54MW10 (L13020654-13), 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.26899837
b = -0.1564446
Y = Area = 24.34469

X = 91.08

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (91.08 * 10 * 1) / (910) = 1.00 µg/L (Signal #1)

Reported Value = 1.00 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW09	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/27/2013 15:08
Collect Date:	02/20/2013 08:40	**Dilution:**	1	**File ID:**	5L009180.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	95.0	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54TM09	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/27/2013 15:47
Collect Date:	02/20/2013 08:40	**Dilution:**	1	**File ID:**	5L009181.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.08	0.269
2,4-Dinitrotoluene	121-14-2		U	1.08	0.269
2,6-Dinitrotoluene	606-20-2		U	1.08	0.269
2,4,6-Trinitrotoluene	118-96-7		U	1.08	0.269
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.08	0.269
2-Nitrotoluene	88-72-2		U	1.08	0.269
4-Nitrotoluene	99-99-0		U	1.08	0.269
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.08	0.269
RDX	121-82-4		U	1.08	0.269
Nitroglycerin	55-63-0		U	1.08	0.269

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	80.2	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-05
Client ID: 54MW11
Matrix: Water
Workgroup #: WG422133
Collect Date: 02/20/2013 10:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: METHOD
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 02/27/2013 08:21
Cal Date: 02/23/2012 15:43
Run Date: 02/27/2013 16:27
File ID: 5L009182.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	106	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-07	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW08	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/27/2013 17:06
Collect Date:	02/20/2013 11:05	**Dilution:**	1	**File ID:**	5L009183.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	109	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	113	50	150	

Sample #: L13020654-13
Client ID: 54MW10
Matrix: Water
Workgroup #: WG422133
Collect Date: 02/20/2013 12:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: METHOD
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 02/27/2013 08:21
Cal Date: 02/23/2012 15:43
Run Date: 02/27/2013 19:03
File ID: 5L009186.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7	1.88	B	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2	0.800	J J	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0	1.00	J J	1.10	0.275
RDX	121-82-4	2.36		1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	93.1	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-13	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW10	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/28/2013 14:43
Collect Date:	02/20/2013 12:35	**Dilution:**	1	**File ID:**	4L024834.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7	1.81	B	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2	0.832	J J	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0	0.737	J J	1.10	0.275
RDX	121-82-4	1.66		1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	93.4	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-15	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW05	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/27/2013 19:42
Collect Date:	02/20/2013 13:35	**Dilution:**	1	**File ID:**	5L009187.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7		U	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.10	0.275
RDX	121-82-4		U	1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	86.6	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-17	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW03	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/27/2013 22:37
Collect Date:	02/20/2013 14:25	**Dilution:**	1	**File ID:**	5L009192.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.08	0.269
2,4-Dinitrotoluene	121-14-2		U	1.08	0.269
2,6-Dinitrotoluene	606-20-2		U	1.08	0.269
2,4,6-Trinitrotoluene	118-96-7		U	1.08	0.269
2-Amino-4,6-dinitrotoluene	35572-78-2	0.367	J	1.08	0.269
2-Nitrotoluene	88-72-2		U	1.08	0.269
4-Nitrotoluene	99-99-0		U	1.08	0.269
4-Amino-2,6-dinitrotoluene	19406-51-0	0.378	J	1.08	0.269
RDX	121-82-4		U	1.08	0.269
Nitroglycerin	55-63-0		U	1.08	0.269

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	91.8	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-17	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW03	**Prep Method:**	METHOD	**Prep Date:**	02/27/2013 08:21
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG422133	**Analyst:**	ECL	**Run Date:**	02/28/2013 15:36
Collect Date:	02/20/2013 14:25	**Dilution:**	1	**File ID:**	4L024835.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.08	0.269
2,4-Dinitrotoluene	121-14-2		U	1.08	0.269
2,6-Dinitrotoluene	606-20-2		U	1.08	0.269
2,4,6-Trinitrotoluene	118-96-7		U	1.08	0.269
2-Amino-4,6-dinitrotoluene	35572-78-2	0.344	J	1.08	0.269
2-Nitrotoluene	88-72-2		U	1.08	0.269
4-Nitrotoluene	99-99-0		U	1.08	0.269
4-Amino-2,6-dinitrotoluene	19406-51-0	0.346	J	1.08	0.269
RDX	121-82-4		U	1.08	0.269
Nitroglycerin	55-63-0		U	1.08	0.269

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	93.4	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13020654

DATE: June 19, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 20, 2013. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of eight aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L13020654-01	54MW08	L13020654-07
54TM09	L13020654-03	54MW10	L13020654-13
54MW11	L13020654-05	54MW05	L13020654-15
54MW11	L13020654-05DL	54MW03	L13020654-17

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications, except for the following. For sample 54MW11 (L13020654-05), sulfate exceeded calibration range. Samples 54MW11 (L13020654-05DL) should be used for this compound. There were no impacts for data usability based on this outlier.

Eric Malarek, Chemist

6/19/13
Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13020654

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/20/13, the coolers were received on 02/21/13 by the primary laboratory (Microbac Laboratories, Inc.) at 2.0°C, 0.0°C, and 1.0°C. Even though the receipt temperatures were below criteria for the coolers; there were no impacts to the data quality and no qualifiers were applied based upon these outliers.

- Holding Time Review: Samples were collected on 02/20/13. The samples were prepped on 02/21/13 and analyzed on 02/21/13 and 02/22/13 for chloride, sulfate, and nitrate analysis, respectively. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 11/29/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW11 (L13020654-05DL), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
02/21/13	Chloride	ICB/CCBs	<LOD	NA	None
02/21/13	Sulfate	ICB/CCBs	<LOD	NA	None
02/21/13	Nitrate	ICB/CCBs	<LOD	NA	None
02/22/13	Chloride	ICB/CCBs	<LOD	NA	None
02/22/13	Sulfate	ICB/CCBs	<LOD	NA	None
02/22/13	Nitrate	ICB/CCBs	<LOD	NA	None
02/21/13	Chloride	WG421668-01	<½MRL	NA	None
02/21/13	Sulfate	WG421668-01	<½MRL	NA	None
02/21/13	Nitrate	WG421668-01	<½MRL	NA	None
02/22/13	Chloride	54RB22113	<½MRL	NA	None
02/22/13	Sulfate	54RB22113	<½MRL	NA	None
02/22/13	Nitrate	54RB22113	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG421668-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 02/21/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW11 (L13020654-05DL), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this LCS.

V-Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- Sample 54MW08 (L13020654-07) was used as the aqueous MS/MSD for the perchlorate analysis on 03/01/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13020654-01), 54TM09 (L13020654-03), 54MW11 (L13020654-05), 54MW11 (L13020654-05DL), 54MW08 (L13020654-07), 54MW10 (L13020654-13), 54MW05 (L13020654-15), and 54MW03 (L13020654-17) apply to this MS/MSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW09 (L13020654-01) and 54TM09 (L13020654-03) was collected for chloride, sulfate, and nitrate analysis. Chloride was detected in the parent sample at 5.54 mg/L and in the duplicate pair at 5.65 mg/L; resulting in a RPD of 2.0%. Sulfate was detected in the parent sample at 117 mg/L and in the duplicate pair at 115 mg/L; resulting in a RPD of 1.7%. Nitrate was detected in the parent sample at 0.510J mg/L and in the duplicate pair at 0.489J mg/L; resulting in a RPD of 4.2%. All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- For sample 54MW11 (L13020654-05), sulfate exceeded calibration range. Samples 54MW11 (L13020654-05DL) should be used for this compound. There were no impacts for data usability based on this outlier.

Sample: 54MW09 (L13020654-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept

DF = Dilution Factor

Given:
m = 0.1331
b = -0.0222
Y = Area = 15.528
DF = 1

X = 116.83 mg/L * DF = 116.83 mg/L * 1 = 117 mg/L

Reported concentration = 117 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-01	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW09	**Prep Method:**	9056	**Prep Date:**	02/21/2013 17:17
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421668	**Analyst:**	JBK	**Run Date:**	02/21/2013 18:24
Collect Date:	02/20/2013 08:40	**Dilution:**	1	**File ID:**	I1_022113-07
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.54		0.200	0.100
Nitrate	14797-55-8	0.510	J J	0.600	0.100
Sulfate	14808-79-8	117		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-03	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54TM09	**Prep Method:**	9056	**Prep Date:**	02/21/2013 17:17
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421668	**Analyst:**	JBK	**Run Date:**	02/21/2013 18:41
Collect Date:	02/20/2013 08:40	**Dilution:**	1	**File ID:**	I1_022113-08
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.65		0.200	0.100
Nitrate	14797-55-8	0.489	J J	0.600	0.100
Sulfate	14808-79-8	115		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-05	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW11	**Prep Method:**	9056	**Prep Date:**	02/21/2013 17:17
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421668	**Analyst:**	JBK	**Run Date:**	02/21/2013 18:59
Collect Date:	02/20/2013 10:00	**Dilution:**	1	**File ID:**	I1_022113-09
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.61		0.200	0.100
Nitrate	14797-55-8	0.346	J J	0.600	0.100
Sulfate	14808-79-8	148	J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.
J	Estimated value ; the analyte concentration was greater than the highest standard

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Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-05	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW11	**Prep Method:**	9056	**Prep Date:**	02/21/2013 17:17
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421668	**Analyst:**	JBK	**Run Date:**	02/22/2013 08:31
Collect Date:	02/20/2013 10:00	**Dilution:**	5	**File ID:**	I1_022113-28
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.05		1.00	0.500
Nitrate	14797-55-8		U	3.00	0.500
Sulfate	14808-79-8	120		5.00	2.50

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020654-07	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW08	**Prep Method:**	9056	**Prep Date:**	02/21/2013 17:17
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421668	**Analyst:**	JBK	**Run Date:**	02/21/2013 19:16
Collect Date:	02/20/2013 11:05	**Dilution:**	1	**File ID:**	I1_022113-10
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.04		0.200	0.100
Nitrate	14797-55-8	0.336	J J	0.600	0.100
Sulfate	14808-79-8	44.6		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-09
Client ID: 54MW08-MS
Matrix: Water
Workgroup #: WG421668
Collect Date: 02/20/2013 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 02/21/2013 17:17
Cal Date: 11/29/2012 10:40
Run Date: 02/21/2013 19:51
File ID: I1_022113-12

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	12.4		0.200	0.100
Nitrate	14797-55-8	5.55		0.600	0.100
Sulfate	14808-79-8	86.9		1.00	0.500

Sample #: L13020654-11
Client ID: 54MW08-MSD
Matrix: Water
Workgroup #: WG421668
Collect Date: 02/20/2013 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 02/21/2013 17:17
Cal Date: 11/29/2012 10:40
Run Date: 02/21/2013 20:08
File ID: I1_022113-13

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	12.4		0.200	0.100
Nitrate	14797-55-8	5.58		0.600	0.100
Sulfate	14808-79-8	87.3		1.00	0.500

Sample #: L13020654-13
Client ID: 54MW10
Matrix: Water
Workgroup #: WG421668
Collect Date: 02/20/2013 12:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 02/21/2013 17:17
Cal Date: 11/29/2012 10:40
Run Date: 02/21/2013 20:26
File ID: I1_022113-14

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.53		0.200	0.100
Nitrate	14797-55-8	0.477	J J	0.600	0.100
Sulfate	14808-79-8	70.1		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L13020654
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020654-15
Client ID: 54MW05
Matrix: Water
Workgroup #: WG421668
Collect Date: 02/20/2013 13:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 02/21/2013 17:17
Cal Date: 11/29/2012 10:40
Run Date: 02/21/2013 21:18
File ID: I1_022113-17

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.16		0.200	0.100
Nitrate	14797-55-8	0.636		0.600	0.100
Sulfate	14808-79-8	15.5		1.00	0.500

Sample #: L13020654-17
Client ID: 54MW03
Matrix: Water
Workgroup #: WG421668
Collect Date: 02/20/2013 14:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 02/21/2013 17:17
Cal Date: 11/29/2012 10:40
Run Date: 02/21/2013 21:35
File ID: I1_022113-18

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.41		0.200	0.100
Nitrate	14797-55-8	0.423	J	0.600	0.100
Sulfate	14808-79-8	25.3		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L13020607

DATE: June 11, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 19, 2013. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of twelve aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW06	L13020607-01	54MW04	L13020607-07
54MW06	L13020607-02	54MW04	L13020607-08
54MW01	L13020607-03	54MW07	L13020607-09
54MW01	L13020607-04	54MW07	L13020607-10
54TM01	L13020607-05	54MW14	L13020607-11
54TM01	L13020607-06	54MW14	L13020607-12

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
X		Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/11/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13020607

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/19/13, the coolers were received on 02/20/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C and 2.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The samples were collected on 02/19/13. The TOC and TIC analysis were run on 02/22/13. The DOC and DIC analysis were run on 02/25/13. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC: 1 - blank
5 - standards ($r \geq 0.995$)
ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 02/22/13. The DOC and DIC analysis were run on 02/25/13. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW06 (L13020607-01), 54MW06 (L13020607-02), 54MW01 (L13020607-03), 54MW01 (L13020607-04), 54TM01 (L13020607-05), 54TM01 (L13020607-06), 54MW04 (L13020607-07), 54MW04 (L13020607-08), 54MW07 (L13020607-09), 54MW07 (L13020607-10), 54MW14 (L13020607-11), and 54MW14 (L13020607-12) apply to these initial and continuing calibrations.

III-Blank Analysis
The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-08) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB22113 (L13020681-07) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
02/22/13	TOC	ICB/CCBs	<LOD	NA	None
02/22/13	TOC	WG421588-01	<½MRL	NA	None
02/25/13	DOC	WG421590-01	<½MRL	NA	None
02/23/13	TOC	54RB22113	0.653J	3.27	54MW06, 54MW01, 54TM01, 54MW04, 54MW07, 54MW14
02/23/13	TIC	54RB22113	<½MRL	NA	None
02/25/13	DOC	54RB22113	0.950J	4.75	54MW06, 54MW01, 54TM01, 54MW04, 54MW07, 54MW14
02/25/13	DIC	54RB22113	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG421588-02 and WG421588-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 02/22/13. All criteria were met. No qualifiers were applied. Samples 54MW06 (L13020607-01), 54MW01 (L13020607-03), 54TM01 (L13020607-05), 54MW04 (L13020607-07), 54MW07 (L13020607-09), and 54MW14 (L13020607-11) apply to this LCS/LCSD.

- Samples WG421590-02 and WG421590-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 02/25/13. All criteria were met. No qualifiers were applied. Samples 54MW06 (L13020607-02), 54MW01 (L13020607-04), 54TM01 (L13020607-06), 54MW04 (L13020607-08), 54MW07 (L13020607-10), and 54MW14 (L13020607-12) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- No project aqueous MS/MSD was performed for TOC, TIC, DOC, and DIC analysis dated 02/22-25/12; therefore was not evaluated. See LCS Section IV for accuracy statement.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW01 (L13020607-03) and 54TM01 (L13020607-05) was collected for TOC and TIC analysis. TOC was detected at 1.19 mg/L in the parent sample and at 1.27 mg/L in the duplicate pair resulting in a RPD of 6.5% (within limits). TIC was detected at 25.8 mg/L in the parent sample and at 21.8 mg/L in the duplicate pair resulting in a RPD of 16.8% (within limits). All criteria were met. No qualifiers were applied.

- Field aqueous sample duplicate pair 54MW01 (L13020607-04) and 54TM01 (L13020607-06) was collected for DOC and DIC analysis. DOC was detected at 2.92 mg/L in the parent sample and at 1.16 mg/L in the duplicate pair resulting in a RPD of 86.3% (outside limits). DIC was detected at 56.7 mg/L in the parent sample and at 55.5 mg/L in the duplicate pair resulting in a RPD of 2.1% (within limits). All criteria were met for DIC. DOC was outside limits; therefore, was qualified estimated "J" for the duplicate pair based upon the high RPD.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW06 (L13020607-01), 54MW06 (L13020607-02), 54MW01 (L13020607-03), 54MW01 (L13020607-04), 54TM01 (L13020607-05), 54TM01 (L13020607-06), 54MW04 (L13020607-07), 54MW04 (L13020607-08), 54MW07 (L13020607-09), 54MW07 (L13020607-10), 54MW14 (L13020607-11), and 54MW14 (L13020607-12) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW06	TOC/DOC	1.0; 2.0	1.50	1.95J	23.1	J
54MW06	TIC/DIC	1.0; 2.0	15.1	36.5	58.6	J
54MW01	TOC/DOC	1.0; 2.0	1.19	2.92	59.3	J
54MW01	TIC/DIC	1.0; 2.0	25.8	56.7	54.5	J
54TM01	TOC/DOC	1.0; 2.0	1.27	1.16J	9.5	None
54TM01	TIC/DIC	1.0; 2.0	21.8	55.5	60.7	J
54MW04	TOC/DOC	1.0; 2.0	1.35	1.60J	15.6	J
54MW04	TIC/DIC	1.0; 2.0	24.3	57.4	57.7	J
54MW07	TOC/DOC	1.0; 2.0	1.50	1.95J	23.1	J
54MW07	TIC/DIC	1.0; 2.0	15.1	36.5	58.6	J
54MW14	TOC/DOC	1.0; 2.0	1.46	2.94	50.3	J
54MW14	TIC/DIC	1.0; 2.0	33.6	76.1	55.9	J

Sample: 54MW4 (L12110202-01), TOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 641.1
DF = 1

X = (16.62 mg/L) * 1 = 16.62 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 447.6
DF = 1

X = (15.12 mg/L) * 1 = 15.12 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 16.62 – 15.12 = 1.50 mg/L

Reported Value = 1.50 mg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Microbac

Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW06	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 14:06
Collect Date:	02/19/2013 10:15	**Dilution:**	1	**File ID:**	TC02222013.006
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.50	B	1.00	0.500

Sample #:	L13020607-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW06	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 14:06
Collect Date:	02/19/2013 10:15	**Dilution:**	1	**File ID:**	TC02222013.006
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		15.1	J	1.00	0.500

Sample #:	L13020607-02	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW06	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 13:06
Collect Date:	02/19/2013 10:15	**Dilution:**	2	**File ID:**	TC2252013..006
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		36.5	J	2.00	1.00

Sample #:	L13020607-02	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW06	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 13:06
Collect Date:	02/19/2013 10:15	**Dilution:**	2	**File ID:**	TC2252013..006
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.95	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13020607-03
Client ID: 54MW01
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/19/2013 11:15
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 14:19
File ID: TC02222013.007

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.19	B	1.00	0.500

Sample #: L13020607-03
Client ID: 54MW01
Matrix: Water
Workgroup #: WG421588
Collect Date: 02/19/2013 11:15
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/22/2013 14:19
File ID: TC02222013.007

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		25.8	J	1.00	0.500

Sample #: L13020607-04
Client ID: 54MW01
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/19/2013 11:15
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 13:19
File ID: TC2252013..007

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		56.7	J	2.00	1.00

Sample #: L13020607-04
Client ID: 54MW01
Matrix: Water
Workgroup #: WG421590
Collect Date: 02/19/2013 11:15
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 02/25/2013 13:19
File ID: TC2252013..007

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.92	B	2.00	1.00

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Certificate of Analysis

Sample #:	L13020607-05	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM01	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 14:32
Collect Date:	02/19/2013 11:15	**Dilution:**	1	**File ID:**	TC02222013.008
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		21.8	J	1.00	0.500

Sample #:	L13020607-05	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM01	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 14:32
Collect Date:	02/19/2013 11:15	**Dilution:**	1	**File ID:**	TC02222013.008
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.27	B	1.00	0.500

Sample #:	L13020607-06	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM01	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 13:33
Collect Date:	02/19/2013 11:15	**Dilution:**	2	**File ID:**	TC2252013..008
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		55.5		2.00	1.00

Sample #:	L13020607-06	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM01	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 13:33
Collect Date:	02/19/2013 11:15	**Dilution:**	2	**File ID:**	TC2252013..008
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.16	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Certificate of Analysis

Sample #:	L13020607-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW04	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 14:46
Collect Date:	02/19/2013 12:35	**Dilution:**	1	**File ID:**	TC02222013.009
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.35	B	1.00	0.500

Sample #:	L13020607-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW04	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 14:46
Collect Date:	02/19/2013 12:35	**Dilution:**	1	**File ID:**	TC02222013.009
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		24.3	J	1.00	0.500

Sample #:	L13020607-08	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW04	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 13:47
Collect Date:	02/19/2013 12:35	**Dilution:**	2	**File ID:**	TC2252013..009
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		57.4	J	2.00	1.00

Sample #:	L13020607-08	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW04	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 13:47
Collect Date:	02/19/2013 12:35	**Dilution:**	2	**File ID:**	TC2252013..009
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.60	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-09	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW07	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 14:59
Collect Date:	02/19/2013 13:30	**Dilution:**	1	**File ID:**	TC02222013.010
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		9.98	J	1.00	0.500

Sample #:	L13020607-09	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW07	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 14:59
Collect Date:	02/19/2013 13:30	**Dilution:**	1	**File ID:**	TC02222013.010
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.791	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-10	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW07	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 14:00
Collect Date:	02/19/2013 13:30	**Dilution:**	2	**File ID:**	TC2252013..010
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.41	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Certificate of Analysis

Sample #:	L13020607-10	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW07	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 14:00
Collect Date:	02/19/2013 13:30	**Dilution:**	2	**File ID:**	TC2252013..010
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		22.2	J	2.00	1.00

Sample #:	L13020607-11	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW14	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 15:12
Collect Date:	02/19/2013 14:05	**Dilution:**	1	**File ID:**	TC02222013.011
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.46	B	1.00	0.500

Sample #:	L13020607-11	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW14	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421588	**Analyst:**	BAF	**Run Date:**	02/22/2013 15:12
Collect Date:	02/19/2013 14:05	**Dilution:**	1	**File ID:**	TC02222013.011
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		33.6	J	1.00	0.500

Sample #:	L13020607-12	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW14	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 14:13
Collect Date:	02/19/2013 14:05	**Dilution:**	2	**File ID:**	TC2252013..011
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.94	B	2.00	1.00

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Microbac

Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-12	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW14	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG421590	**Analyst:**	BAF	**Run Date:**	02/25/2013 14:13
Collect Date:	02/19/2013 14:05	**Dilution:**	2	**File ID:**	TC2252013..011
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		76.1	J	2.00	1.00

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13020607

DATE: June 11, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 19, 2013. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of six aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW06	L13020607-01	54MW04	L13020607-07
54MW01	L13020607-03	54MW07	L13020607-09
54TM01	L13020607-05	54MW14	L13020607-11

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/11/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13020607

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/19/13, the coolers were received on 02/20/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C and 2.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 02/19/13 for perchlorate analysis. The aqueous samples were prepped and analyzed on 02/25/13. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check
LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:
1- blank (<1/2MRL DoD Perchlorate Handbook)
5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
ICV (≤15%D DoD Perchlorate Handbook)
CCV/ICS (≤15%D DoD Perchlorate Handbook)
LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 01/29/13 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW06 (L13020607-01), 54MW01 (L13020607-03), 54TM01 (L13020607-05), 54MW04 (L13020607-07), 54MW07 (L13020607-09), and 54MW14 (L13020607-11) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
02/25/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
02/25/13	WG421771-02	All perchlorate <½MRL	NA	NA	None
03/01/13	54RB22113	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG421771-03 was used as aqueous LCS for perchlorate analysis dated 02/25/13. All criteria were met. No qualifiers were applied. Samples 54MW06 (L13020607-01), 54MW01 (L13020607-03), 54TM01 (L13020607-05), 54MW04 (L13020607-07), 54MW07 (L13020607-09), and 54MW14 (L13020607-11) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- No site specific aqueous MS/MSD was analyzed for perchlorate analysis in this SDG; therefore, was not evaluated. See LCS/LCSD Section VI for laboratory accuracy statement.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW01 (L13020607-03) and 54TM01 (L13020607-05) was analyzed for perchlorate analysis in this SDG. Perchlorate was non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW06 (L13020607-01), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.19
b = 0.000928
Y = Area = 11800/304000 = 0.038816
X = 0.03184

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.03184 * 5 * 1) = 0.160 µg/L (Signal #1)

Reported Value = 0.160 µg/L
% Difference = 0.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Microbac

Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-01	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW06	**Prep Method:**	6850	**Prep Date:**	02/25/2013 12:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	01/29/2013 19:25
Workgroup #:	WG421771	**Analyst:**	JWR	**Run Date:**	02/25/2013 20:34
Collect Date:	02/19/2013 10:15	**Dilution:**	1	**File ID:**	1LM.LM19850
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.160	J J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-03	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW01	**Prep Method:**	6850	**Prep Date:**	02/25/2013 12:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	01/29/2013 19:25
Workgroup #:	WG421771	**Analyst:**	JWR	**Run Date:**	02/25/2013 22:09
Collect Date:	02/19/2013 11:15	**Dilution:**	1	**File ID:**	1LM.LM19855
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-05	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54TM01	**Prep Method:**	6850	**Prep Date:**	02/25/2013 12:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	01/29/2013 19:25
Workgroup #:	WG421771	**Analyst:**	JWR	**Run Date:**	02/25/2013 22:28
Collect Date:	02/19/2013 11:15	**Dilution:**	1	**File ID:**	1LM.LM19856
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-07	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW04	**Prep Method:**	6850	**Prep Date:**	02/25/2013 12:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	01/29/2013 19:25
Workgroup #:	WG421771	**Analyst:**	JWR	**Run Date:**	02/25/2013 22:47
Collect Date:	02/19/2013 12:35	**Dilution:**	1	**File ID:**	1LM.LM19857
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-09	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW07	**Prep Method:**	6850	**Prep Date:**	02/25/2013 12:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	01/29/2013 19:25
Workgroup #:	WG421771	**Analyst:**	JWR	**Run Date:**	02/25/2013 23:06
Collect Date:	02/19/2013 13:30	**Dilution:**	1	**File ID:**	1LM.LM19858
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.162	J J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-11	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW14	**Prep Method:**	6850	**Prep Date:**	02/25/2013 12:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	01/29/2013 19:25
Workgroup #:	WG421771	**Analyst:**	JWR	**Run Date:**	02/25/2013 23:25
Collect Date:	02/19/2013 14:05	**Dilution:**	1	**File ID:**	1LM.LM19859
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Eric.malarek@CBI.com

MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13020607

DATE: June 11, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 19, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of six aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW06	L13020607-01	54MW04	L13020607-07
54MW01	L13020607-03	54MW07	L13020607-09
54TM01	L13020607-05	54MW14	L13020607-11

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable.

Eric Malarek, Chemist

6/11/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13020607

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/19/13, the coolers were received on 02/20/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C and 2.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 02/19/13. The samples were extracted on 02/20/13 and were analyzed on 02/21/13 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
02/21/13	WG421324-01	All target explosives <½MRL	NA	NA	None
02/21/13	WG421324-01	All target explosives <½MRL	NA	NA	None
02/28/13	54RB22113	2,4,6-Trinitrotoluene	1.05J	5.25	None

J = Estimated Value <MRL and >MDL.
NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW06 (L13020607-01), 54MW01 (L13020607-03), 54TM01 (L13020607-05), 54MW04 (L13020607-07), 54MW07 (L13020607-09), and 54MW14 (L13020607-11) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. No confirmation samples were required for all associated samples were non-detect. No samples apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 02/21/13 @09:12 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW06 (L13020607-01), 54MW01 (L13020607-03), 54TM01 (L13020607-05), 54MW04 (L13020607-07), 54MW07 (L13020607-09), and 54MW14 (L13020607-11) were reported using this continuing calibration.

- For explosives continuing calibration performed on 02/21/13 @18:03 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample and Laboratory Control Sample Duplicate
Laboratory control samples (LCS) and LCSD are used to monitor long-term precision and accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS/LCSD recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Samples WG421324-02 and WG421324-03 were used as the aqueous LCS/LCSD for the explosives analysis on 02/21/13. All criteria were met. No qualifiers were applied. Samples 54MW06 (L13020607-01), 54MW01 (L13020607-03), 54TM01 (L13020607-05), 54MW04 (L13020607-07), 54MW07 (L13020607-09), and 54MW14 (L13020607-11) apply to this LCS/LCSD.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- No site specific aqueous MS/MSD was analyzed for explosives analysis in this SDG; therefore, was not evaluated. See LCS/LCSD Section VI for laboratory precision and accuracy statements.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW01 (L13020607-03) and 54TM01 (L13020607-05) was analyzed for explosives analysis in this SDG. All target explosives were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

IX-Quantitation Verification and Data Review
The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- All associated samples were non-detect for all target compounds. No further confirmation criterion is required in this SDG.

Sample: WG421324-02, 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.26899837
b = -0.1564446
Y = Area = 110.12210

X = 409.96

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (409.96 * 10 * 1) / (1000) = 4.10 µg/L (Signal #1)

Reported Value = 4.10 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW06	**Prep Method:**	METHOD	**Prep Date:**	02/20/2013 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG421443	**Analyst:**	ECL	**Run Date:**	02/21/2013 12:32
Collect Date:	02/19/2013 10:15	**Dilution:**	1	**File ID:**	5L009153.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.08	0.269
2,4-Dinitrotoluene	121-14-2		U	1.08	0.269
2,6-Dinitrotoluene	606-20-2		U	1.08	0.269
2,4,6-Trinitrotoluene	118-96-7		U	1.08	0.269
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.08	0.269
2-Nitrotoluene	88-72-2		U	1.08	0.269
4-Nitrotoluene	99-99-0		U	1.08	0.269
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.08	0.269
RDX	121-82-4		U	1.08	0.269
Nitroglycerin	55-63-0		U	1.08	0.269

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	93.2	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW01	**Prep Method:**	METHOD	**Prep Date:**	02/20/2013 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG421443	**Analyst:**	ECL	**Run Date:**	02/21/2013 13:11
Collect Date:	02/19/2013 11:15	**Dilution:**	1	**File ID:**	5L009154.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.05	0.263
2,4-Dinitrotoluene	121-14-2		U	1.05	0.263
2,6-Dinitrotoluene	606-20-2		U	1.05	0.263
2,4,6-Trinitrotoluene	118-96-7		U	1.05	0.263
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.05	0.263
2-Nitrotoluene	88-72-2		U	1.05	0.263
4-Nitrotoluene	99-99-0		U	1.05	0.263
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.05	0.263
RDX	121-82-4		U	1.05	0.263
Nitroglycerin	55-63-0		U	1.05	0.263

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	92.0	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54TM01	**Prep Method:**	METHOD	**Prep Date:**	02/20/2013 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG421443	**Analyst:**	ECL	**Run Date:**	02/21/2013 13:50
Collect Date:	02/19/2013 11:15	**Dilution:**	1	**File ID:**	5L009155.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.09	0.272
2,4-Dinitrotoluene	121-14-2		U	1.09	0.272
2,6-Dinitrotoluene	606-20-2		U	1.09	0.272
2,4,6-Trinitrotoluene	118-96-7		U	1.09	0.272
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.09	0.272
2-Nitrotoluene	88-72-2		U	1.09	0.272
4-Nitrotoluene	99-99-0		U	1.09	0.272
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.09	0.272
RDX	121-82-4		U	1.09	0.272
Nitroglycerin	55-63-0		U	1.09	0.272

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	96.6	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-07	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW04	**Prep Method:**	METHOD	**Prep Date:**	02/20/2013 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG421443	**Analyst:**	ECL	**Run Date:**	02/21/2013 14:29
Collect Date:	02/19/2013 12:35	**Dilution:**	1	**File ID:**	5L009156.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	87.6	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW07	**Prep Method:**	METHOD	**Prep Date:**	02/20/2013 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG421443	**Analyst:**	ECL	**Run Date:**	02/21/2013 15:08
Collect Date:	02/19/2013 13:30	**Dilution:**	1	**File ID:**	5L009157.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	2.38	0.595
2,4-Dinitrotoluene	121-14-2		U	2.38	0.595
2,6-Dinitrotoluene	606-20-2		U	2.38	0.595
2,4,6-Trinitrotoluene	118-96-7		U	2.38	0.595
2-Amino-4,6-dinitrotoluene	35572-78-2		U	2.38	0.595
2-Nitrotoluene	88-72-2		U	2.38	0.595
4-Nitrotoluene	99-99-0		U	2.38	0.595
4-Amino-2,6-dinitrotoluene	19406-51-0		U	2.38	0.595
RDX	121-82-4		U	2.38	0.595
Nitroglycerin	55-63-0		U	2.38	0.595

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	65.7	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-11	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW14	**Prep Method:**	METHOD	**Prep Date:**	02/20/2013 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG421443	**Analyst:**	ECL	**Run Date:**	02/21/2013 15:47
Collect Date:	02/19/2013 14:05	**Dilution:**	1	**File ID:**	5L009158.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.09	0.272
2,4-Dinitrotoluene	121-14-2		U	1.09	0.272
2,6-Dinitrotoluene	606-20-2		U	1.09	0.272
2,4,6-Trinitrotoluene	118-96-7		U	1.09	0.272
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.09	0.272
2-Nitrotoluene	88-72-2		U	1.09	0.272
4-Nitrotoluene	99-99-0		U	1.09	0.272
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.09	0.272
RDX	121-82-4		U	1.09	0.272
Nitroglycerin	55-63-0		U	1.09	0.272

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	95.7	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13020607

DATE: June 11, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on February 19, 2013. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of six aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW06	L13020607-01	54MW04	L13020607-07
54MW01	L13020607-03	54MW07	L13020607-09
54TM01	L13020607-05	54MW14	L13020607-11

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
X		Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/11/13
Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13020607

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 02/19/13, the coolers were received on 02/20/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C and 2.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: Samples were collected on 02/19/13. The samples were prepped on 02/20/13 and 02/21/13 and analyzed on 02/20/13 and 02/22/13 for chloride, sulfate, and nitrate analysis, respectively. Sample collection dates may be found on the attached form 1s. Sample 54MW07 (L13020607-09) was analyzed one day past prescribed holding times for nitrate and was qualified estimated bias low "L" based on this outlier. For all other analytes and samples, all criteria were met.

II-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 11/29/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW06 (L13020607-01), 54MW01 (L13020607-03), 54TM01 (L13020607-05), 54MW04 (L13020607-07), 54MW07 (L13020607-09), and 54MW14 (L13020607-11) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB22113 (L13020681-07) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
02/20/13	Chloride	ICB/CCBs	<LOD	NA	None
02/20/13	Sulfate	ICB/CCBs	<LOD	NA	None
02/20/13	Nitrate	ICB/CCBs	<LOD	NA	None
02/22/13	Chloride	ICB/CCBs	<LOD	NA	None
02/22/13	Sulfate	ICB/CCBs	<LOD	NA	None
02/22/13	Nitrate	ICB/CCBs	<LOD	NA	None
02/20/13	Chloride	WG421411-01	<½MRL	NA	None
02/20/13	Sulfate	WG421411-01	<½MRL	NA	None
02/20/13	Nitrate	WG421411-01	<½MRL	NA	None
02/21/13	Chloride	WG421668-01	<½MRL	NA	None
02/21/13	Sulfate	WG421668-01	<½MRL	NA	None
02/21/13	Nitrate	WG421668-01	<½MRL	NA	None
02/22/13	Chloride	54RB22113	<½MRL	NA	None
02/22/13	Sulfate	54RB22113	<½MRL	NA	None
02/22/13	Nitrate	54RB22113	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG421411-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 02/20/13. All criteria were met. No qualifiers were applied. Samples 54MW06 (L13020607-01), 54MW01 (L13020607-03), 54TM01 (L13020607-05), 54MW04 (L13020607-07), and 54MW14 (L13020607-11) apply to this LCS.

- Sample WG421668-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 02/21/13. All criteria were met. No qualifiers were applied. Sample 54MW07 (L13020607-09) applies to this LCS.

V-Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- No project aqueous MS/MSD was performed for chloride, sulfate, and nitrate analysis dated 11/08/12; therefore was not evaluated. See LCS Section IV for accuracy statement.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW01 (L13020607-03) and 54TM01 (L13020607-05) was collected for chloride, sulfate, and nitrate analysis. Chloride was detected in the parent sample at 1.25 mg/L and in the duplicate pair at 1.26 mg/L; resulting in a RPD of 0.8%. Sulfate was detected in the parent sample at 27.1 mg/L and in the duplicate pair at 27.0 mg/L; resulting in a RPD of 0.4%. Nitrate was not detected in the duplicate pair. All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW06 (L13020607-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1331
b = -0.0222
Y = Area = 0.549
DF = 1

X = 4.29 mg/L * DF = 4.29 mg/L * 1 = 4.29 mg/L

Reported concentration = 4.29 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-01	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW06	**Prep Method:**	9056	**Prep Date:**	02/20/2013 14:12
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421411	**Analyst:**	JBK	**Run Date:**	02/20/2013 15:19
Collect Date:	02/19/2013 10:15	**Dilution:**	1	**File ID:**	I1_022013-07
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	0.680		0.200	0.100
Nitrate	14797-55-8	0.209	J J	0.600	0.100
Sulfate	14808-79-8	4.29		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-03	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW01	**Prep Method:**	9056	**Prep Date:**	02/20/2013 14:12
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421411	**Analyst:**	JBK	**Run Date:**	02/20/2013 15:36
Collect Date:	02/19/2013 11:15	**Dilution:**	1	**File ID:**	I1_022013-08
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.25		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	27.1		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-05	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54TM01	**Prep Method:**	9056	**Prep Date:**	02/20/2013 14:12
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421411	**Analyst:**	JBK	**Run Date:**	02/20/2013 15:53
Collect Date:	02/19/2013 11:15	**Dilution:**	1	**File ID:**	I1_022013-09
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.26		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	27.0		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-07	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW04	**Prep Method:**	9056	**Prep Date:**	02/20/2013 14:12
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421411	**Analyst:**	JBK	**Run Date:**	02/20/2013 16:11
Collect Date:	02/19/2013 12:35	**Dilution:**	5	**File ID:**	I1_022013-10
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.96		1.00	0.500
Nitrate	14797-55-8		U	3.00	0.500
Sulfate	14808-79-8	340		5.00	2.50

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-09	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW07	**Prep Method:**	9056	**Prep Date:**	02/21/2013 17:17
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421668	**Analyst:**	JBK	**Run Date:**	02/22/2013 08:49
Collect Date:	02/19/2013 13:30	**Dilution:**	1	**File ID:**	I1_022113-29
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.75		0.200	0.100
Nitrate	14797-55-8	0.205	J J	0.600	0.100
Sulfate	14808-79-8	34.2		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L13020607
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13020607-11	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW14	**Prep Method:**	9056	**Prep Date:**	02/20/2013 14:12
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	11/29/2012 10:40
Workgroup #:	WG421411	**Analyst:**	JBK	**Run Date:**	02/20/2013 16:46
Collect Date:	02/19/2013 14:05	**Dilution:**	1	**File ID:**	I1_022013-12
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.37		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	41.1		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L12110249

DATE: June 7, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 8, 2012. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions, except for samples 54RB110812, 54MW2, and 54MW12. For 54RB110812, 54MW2, and 54MW12, the samples with the odd fraction lab IDs are the dissolved fractions and the samples with the even fraction lab IDs are the total fractions. A total of fifteen aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW1	L12110249-01	TOC CK	L12110249-13
54MW1	L12110249-02	54RB110812	L12110249-14
54MW5	L12110249-07	54RB110812	L12110249-15
54MW5	L12110249-08	54MW2	L12110249-16
54MW14	L12110249-09	54MW2	L12110249-17
54MW14	L12110249-10	54MW12	L12110249-18
54MW3	L12110249-11	54MW12	L12110249-19
54MW3	L12110249-12		

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
X		Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/7/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L12110249

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/08/12, the coolers were received on 11/09/12 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C, 0.0°C, and 1.0°C. Even though two of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The samples were collected on 11/08/12. The TOC and TIC analysis were run on 11/12/12. The DOC and DIC analysis were run on 11/12/12 and 11/13/12. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC:	1 - blank
	5 - standards ($r \geq 0.995$)
	ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 11/12/12. The DOC and DIC analysis were run on 11/12/12 and 11/13/12. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW1 (L12110249-02), 54MW5 (L12110249-07), 54MW5 (L12110249-08), 54MW14 (L12110249-09), 54MW14 (L12110249-10), 54MW3 (L12110249-11), 54MW3 (L12110249-12), TOC CK (L12110249-13), 54RB110812 (L12110249-14), 54RB110812 (L12110249-15), 54MW2 (L12110249-16), 54MW2 (L12110249-17), 54MW12 (L12110249-18), and 54MW12 (L12110249-19) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-15) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB110812 (L12110249-14) applies to all total samples (TOC and TIC) in this SDG. The decontamination water blank TOC CK (L12110249-13) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
11/12/12	TOC	ICB/CCBs	<LOD	NA	None
11/12/12	TOC	WG413955-01	<½MRL	NA	None
11/13/12	DOC	WG413963-01	<½MRL	NA	None
11/12/12	TOC	54RB110812	0.675J	3.38	54MW1, 54MW14, 54MW3, 54MW2
11/12/12	TIC	54RB110812	<½MRL	NA	None
11/13/12	DOC	54RB110812	1.62	8.10	54MW5, 54MW14, 54MW3, 54MW12
11/13/12	DIC	54RB110812	<½MRL	NA	None
11/12/12	TOC	TOC CK	0.778J	3.89	54MW1, 54MW14, 54MW3, 54MW2, 54MW12
11/12/12	TIC	TOC CK	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG413955-02 and WG413955-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 11/12/12. All criteria were met. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), TOC CK (L12110249-13), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this LCS/LCSD.

- Samples WG413963-02 and WG413963-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 11/12/12. All criteria were met. No qualifiers were applied. Samples 54MW1 (L12110249-02), 54MW5 (L12110249-08), 54MW14 (L12110249-10), 54MW3 (L12110249-12), 54RB110812 (L12110249-15), 54MW2 (L12110249-17), and 54MW12 (L12110249-19) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate
Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- Sample 54MW10 (L12010471-09) was used as the aqueous MS/MSD for TOC and TIC analysis on 01/23/12. All criteria were met. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), TOC CK (L12110249-13), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this MS/MSD.

- Sample 54MW10 (L12010471-10) was used as the aqueous MS/MSD for DOC and DIC analysis on 01/24/12. DOC (RPD=18.3%) was outside criteria. DOC and DIC were qualified estimated "J" for detections based upon the high RPD. Samples 54MW1 (L12110249-02), 54MW5 (L12110249-08), 54MW14 (L12110249-10), 54MW3 (L12110249-12), 54RB110812 (L12110249-15), 54MW2 (L12110249-17), and 54MW12 (L12110249-19) apply to this MS/MSD.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG; therefore, was not evaluated.

VIII-Quantitation Verification and Data Review

The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW1 (L12110249-01), 54MW1 (L12110249-02), 54MW5 (L12110249-07), 54MW5 (L12110249-08), 54MW14 (L12110249-09), 54MW14 (L12110249-10), 54MW3 (L12110249-11), 54MW3 (L12110249-12), 54RB110812 (L12110249-14), 54RB110812 (L12110249-15), 54MW2 (L12110249-16), 54MW2 (L12110249-17), 54MW12 (L12110249-18), and 54MW12 (L12110249-19) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW1	TOC/DOC	1.0; 2.0	1.96	2.00U	NA	None
54MW1	TIC/DIC	1.0; 2.0	25.0	55.0	54.6	J
54MW5	TOC/DOC	1.0; 2.0	4.31	7.39	41.7	J
54MW5	TIC/DIC	1.0; 2.0	41.8	81.2	48.5	J
54MW14	TOC/DOC	1.0; 3.0	2.12	6.58	67.8	J
54MW14	TIC/DIC	1.0; 3.0	33.9	75.7	55.2	J
54MW3	TOC/DOC	1.0; 2.0	3.29	7.65	57.0	J
54MW3	TIC/DIC	1.0; 2.0	33.6	71.8	53.2	J
54RB110812	TOC/DOC	1.0; 1.0	0.675J	1.62	58.3	J
54RB110812	TIC/DIC	1.0; 1.0	1.00U	1.00U	NA	None
54MW2	TOC/DOC	1.0; 2.0	2.68	8.99	70.2	J
54MW2	TIC/DIC	1.0; 2.0	29.1	82.1	64.6	J
55MW12	TOC/DOC	1.0; 2.0	3.41	7.88	56.7	J
54MW12	TIC/DIC	1.0; 2.0	27.9	66.1	57.8	J

J = Estimated value.
U = Non-detect as <MRL.
MRL = Method Reporting Limit

Sample: 54MW1 (L12110249-01), TOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 1037
DF = 1
X = (26.91 mg/L) * 1 = 26.91 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 734.2
DF = 1
X = (24.95 mg/L) * 1 = 24.95 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 26.91 – 24.95 = 1.96 mg/L

Reported Value = 1.96 mg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-01
Client ID: 54MW1
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 15:11
File ID: TC11122012.017

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		25.0	J	1.00	0.500

Sample #: L12110249-01
Client ID: 54MW1
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 15:11
File ID: TC11122012.017

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.96	B	1.00	0.500

Sample #: L12110249-02
Client ID: 54MW1
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/08/2012 07:45
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 10:09
File ID: TC11122012.091

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC		U	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-02
Client ID: 54MW1
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/08/2012 07:45
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 10:09
File ID: TC11122012.091

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		55.0	J	2.00	1.00

Sample #: L12110249-03
Client ID: 54MW1
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 15:40
File ID: TC11122012.019

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	12.5		1.00	0.500

Sample #: L12110249-03
Client ID: 54MW1
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 15:40
File ID: TC11122012.019

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		14.0		1.00	0.500

Sample #: L12110249-04
Client ID: 54MW1
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/08/2012 07:45
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 10:36
File ID: TC11122012.093

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		47.7		2.00	1.00

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Certificate of Analysis

Sample #: L12110249-06 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW1 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 | **Analyst:** DIH | **Run Date:** 11/13/2012 10:50
Collect Date: 11/08/2012 07:45 | **Dilution:** 2 | **File ID:** TC11122012.094
Sample Tag: DL01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
~~Organic Carbon, Dissolved~~	~~TOC~~	~~11.0~~		~~2.00~~	~~1.00~~

Sample #: L12110249-07 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW5 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 | **Analyst:** DIH | **Run Date:** 11/12/2012 16:08
Collect Date: 11/08/2012 09:50 | **Dilution:** 1 | **File ID:** TC11122012.021
Sample Tag: 01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		41.8	J	1.00	0.500

Sample #: L12110249-07 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW5 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 | **Analyst:** DIH | **Run Date:** 11/12/2012 16:08
Collect Date: 11/08/2012 09:50 | **Dilution:** 1 | **File ID:** TC11122012.021
Sample Tag: 01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	4.31	J	1.00	0.500

Sample #: L12110249-08 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW5 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 | **Analyst:** DIH | **Run Date:** 11/12/2012 23:41
Collect Date: 11/08/2012 09:50 | **Dilution:** 2 | **File ID:** TC11122012.053
Sample Tag: DL01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	7.39	B	2.00	1.00

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Certificate of Analysis

Sample #: L12110249-08 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW5 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 **Analyst:** DIH **Run Date:** 11/12/2012 23:41
Collect Date: 11/08/2012 09:50 **Dilution:** 2 **File ID:** TC11122012.053
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		81.2	J	2.00	1.00

Sample #: L12110249-09 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW14 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 **Analyst:** DIH **Run Date:** 11/12/2012 16:22
Collect Date: 11/08/2012 11:05 **Dilution:** 1 **File ID:** TC11122012.022
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.12	B	1.00	0.500

Sample #: L12110249-09 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW14 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 **Analyst:** DIH **Run Date:** 11/12/2012 16:22
Collect Date: 11/08/2012 11:05 **Dilution:** 1 **File ID:** TC11122012.022
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		33.9	J	1.00	0.500

Sample #: L12110249-10 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW14 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 **Analyst:** DIH **Run Date:** 11/12/2012 23:56
Collect Date: 11/08/2012 11:05 **Dilution:** 2 **File ID:** TC11122012.054
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	6.58	B	2.00	1.00

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-10 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW14 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 | **Analyst:** DIH | **Run Date:** 11/12/2012 23:56
Collect Date: 11/08/2012 11:05 | **Dilution:** 2 | **File ID:** TC11122012.054
Sample Tag: DL01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		75.7	J	2.00	1.00

Sample #: L12110249-11 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW3 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 | **Analyst:** DIH | **Run Date:** 11/12/2012 16:37
Collect Date: 11/08/2012 12:15 | **Dilution:** 1 | **File ID:** TC11122012.023
Sample Tag: 01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.29	B	1.00	0.500

Sample #: L12110249-11 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW3 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 | **Analyst:** DIH | **Run Date:** 11/12/2012 16:37
Collect Date: 11/08/2012 12:15 | **Dilution:** 1 | **File ID:** TC11122012.023
Sample Tag: 01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		33.6	J	1.00	0.500

Sample #: L12110249-12 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW3 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 | **Analyst:** DIH | **Run Date:** 11/13/2012 00:12
Collect Date: 11/08/2012 12:15 | **Dilution:** 2 | **File ID:** TC11122012.055
Sample Tag: DL01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		71.8	J	2.00	1.00

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-12
Client ID: 54MW3
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/08/2012 12:15
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 00:12
File ID: TC11122012.055

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	7.65	B	2.00	1.00

Sample #: L12110249-13
Client ID: TOC CK
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/08/2012 11:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 16:50
File ID: TC11122012.024

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.778	J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-13	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	TOC CK	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG413955	**Analyst:**	DIH	**Run Date:**	11/12/2012 16:50
Collect Date:	11/08/2012 11:45	**Dilution:**	1	**File ID:**	TC11122012.024
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-14
Client ID: 54RB110812
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/08/2012 12:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 17:02
File ID: TC11122012.025

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-14	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB110812	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG413955	**Analyst:**	DIH	**Run Date:**	11/12/2012 17:02
Collect Date:	11/08/2012 12:45	**Dilution:**	1	**File ID:**	TC11122012.025
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.675	J J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-15	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB110812	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG413963	**Analyst:**	DIH	**Run Date:**	11/13/2012 00:25
Collect Date:	11/08/2012 12:45	**Dilution:**	1	**File ID:**	TC11122012.056
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Certificate of Analysis

Sample #: L12110249-15 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54RB110812 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 **Analyst:** DIH **Run Date:** 11/13/2012 00:25
Collect Date: 11/08/2012 12:45 **Dilution:** 1 **File ID:** TC11122012.056
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.62	J	1.00	0.500

Sample #: L12110249-16 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW2 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 **Analyst:** DIH **Run Date:** 11/12/2012 17:38
Collect Date: 11/08/2012 13:45 **Dilution:** 1 **File ID:** TC11122012.028
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		29.1	J	1.00	0.500

Sample #: L12110249-16 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW2 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 **Analyst:** DIH **Run Date:** 11/12/2012 17:38
Collect Date: 11/08/2012 13:45 **Dilution:** 1 **File ID:** TC11122012.028
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.68	B	1.00	0.500

Sample #: L12110249-17 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW2 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 **Analyst:** DIH **Run Date:** 11/13/2012 00:40
Collect Date: 11/08/2012 13:45 **Dilution:** 2 **File ID:** TC11122012.057
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		82.1	J	2.00	1.00

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-17
Client ID: 54MW2
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/08/2012 13:45
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 00:40
File ID: TC11122012.057

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	8.99	J	2.00	1.00

Sample #: L12110249-18
Client ID: 54MW12
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/08/2012 14:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 17:53
File ID: TC11122012.029

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		27.9	J	1.00	0.500

Sample #: L12110249-18
Client ID: 54MW12
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/08/2012 14:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 17:53
File ID: TC11122012.029

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.41	B	1.00	0.500

Sample #: L12110249-19
Client ID: 54MW12
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/08/2012 14:55
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 00:55
File ID: TC11122012.058

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	7.88	B	2.00	1.00

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-19	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW12	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG413963	**Analyst:**	DIH	**Run Date:**	11/13/2012 00:55
Collect Date:	11/08/2012 14:55	**Dilution:**	2	**File ID:**	TC11122012.058
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		66.1	J	2.00	1.00

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L12110249

DATE: June 6, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 8, 2012. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of seven aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW1	L12110249-01	54RB110812	L12110249-14
54MW5	L12110249-07	54MW2	L12110249-16
54MW14	L12110249-09	54MW12	L12110249-18
54MW3	L12110249-11		

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable.

Eric Malarek, Chemist

6/6/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L12110249

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/08/12, the coolers were received on 11/09/12 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C, 0.0°C, and 1.0°C. Even though two of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 11/08/12 for perchlorate analysis. The aqueous samples were prepped on 11/19/12 and analyzed on 11/19/12 and 11/20/12. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check

LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:	1- blank (<1/2MRL DoD Perchlorate Handbook)
	5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
	ICV (≤15%D DoD Perchlorate Handbook)
	CCV/ICS (≤15%D DoD Perchlorate Handbook)
	LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 11/19/12 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
11/19/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
11/20/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
11/19/12	WG414690-02	All perchlorate <½MRL	NA	NA	None
11/20/12	54RB110812	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG414690-03 was used as aqueous LCS for perchlorate analysis dated 11/19/12. All criteria were met. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- Sample 54MW1 (L12110249-01) was used as the aqueous MS/MSD for the perchlorate analysis on 11/19/12. All criteria were met. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this MS/MSD.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for explosives analysis in this SDG; therefore, was not evaluated.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW5 (L12110249-07), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.27
b = 0.00313
Y = Area = 20400/274000 = 0.07445
X = 0.05616

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.05616 * 5 * 1) = 0.280 µg/L (Signal #1)
Reported Value = 0.280 µg/L
% Difference = 0.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-01	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW1	**Prep Method:**	6850	**Prep Date:**	11/19/2012 17:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	11/19/2012 19:00
Workgroup #:	WG414690	**Analyst:**	JWR	**Run Date:**	11/19/2012 21:51
Collect Date:	11/08/2012 07:45	**Dilution:**	1	**File ID:**	1LM.LM18695
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-03
Client ID: 54MW1
Matrix: Water
Workgroup #: WG414690
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/19/2012 17:00
Cal Date: 11/19/2012 19:00
Run Date: 11/19/2012 22:10
File ID: 1LM.LM18696

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.210		0.200	0.100

Sample #: L12110249-05
Client ID: 54MW1
Matrix: Water
Workgroup #: WG414690
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/19/2012 17:00
Cal Date: 11/19/2012 19:00
Run Date: 11/19/2012 22:29
File ID: 1LM.LM18697

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.205		0.200	0.100

Sample #: L12110249-07
Client ID: 54MW5
Matrix: Water
Workgroup #: WG414690
Collect Date: 11/08/2012 09:50
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/19/2012 17:00
Cal Date: 11/19/2012 19:00
Run Date: 11/19/2012 22:47
File ID: 1LM.LM18698

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.280		0.200	0.100

Sample #: L12110249-09
Client ID: 54MW14
Matrix: Water
Workgroup #: WG414690
Collect Date: 11/08/2012 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/19/2012 17:00
Cal Date: 11/19/2012 19:00
Run Date: 11/19/2012 23:06
File ID: 1LM.LM18699

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-11 **PrePrep Method:** N/A **Instrument:** LCMS1
Client ID: 54MW3 **Prep Method:** 6850 **Prep Date:** 11/19/2012 17:00
Matrix: Water **Analytical Method:** 6850 **Cal Date:** 11/19/2012 19:00
Workgroup #: WG414690 **Analyst:** JWR **Run Date:** 11/20/2012 00:41
Collect Date: 11/08/2012 12:15 **Dilution:** 1 **File ID:** 1LM.LM18704
Sample Tag: 01 **Units:** ug/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.303		0.200	0.100

Sample #: L12110249-14 **PrePrep Method:** N/A **Instrument:** LCMS1
Client ID: 54RB110812 **Prep Method:** 6850 **Prep Date:** 11/19/2012 17:00
Matrix: Water **Analytical Method:** 6850 **Cal Date:** 11/19/2012 19:00
Workgroup #: WG414690 **Analyst:** JWR **Run Date:** 11/20/2012 01:00
Collect Date: 11/08/2012 12:45 **Dilution:** 1 **File ID:** 1LM.LM18705
Sample Tag: 01 **Units:** ug/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-16
Client ID: 54MW2
Matrix: Water
Workgroup #: WG414690
Collect Date: 11/08/2012 13:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/19/2012 17:00
Cal Date: 11/19/2012 19:00
Run Date: 11/20/2012 01:19
File ID: 1LM.LM18706

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.842		0.200	0.100

Sample #: L12110249-18
Client ID: 54MW12
Matrix: Water
Workgroup #: WG414690
Collect Date: 11/08/2012 14:55
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/19/2012 17:00
Cal Date: 11/19/2012 19:00
Run Date: 11/20/2012 01:38
File ID: 1LM.LM18707

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	5.31		0.200	0.100

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L12110249

DATE: June 5, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 8, 2012. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of nine aqueous samples (includes two re-analysis samples) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW1	L12110249-01	54RB110812	L12110249-14
54MW5	L12110249-07	54MW2	L12110249-16
54MW14	L12110249-09	54MW12	L12110249-18
54MW3	L12110249-11	54MW12	L12110249-18RE
54MW3	L12110249-11RE		

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

6/5/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L12110249

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/08/12, the coolers were received on 11/09/12 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C, 0.0°C, and 1.0°C. Even though two of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 11/08/12. The samples were extracted on 11/14/12 and were analyzed on 11/14/12 and 11/16/12 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
11/14/12	WG414169-02	All target explosives <½MRL	NA	NA	None
11/16/12	WG414169-02	All target explosives <½MRL	NA	NA	None
11/14/12	54RB110812	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Confirmation samples 54MW3 (L12110249-11RE) and 54MW12 (L12110249-18RE) apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 11/14/12 @11:05 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), and 54RB110812 (L12110249-14) were reported using this continuing calibration.

- For explosives continuing calibration performed on 11/14/12 @19:57 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW2 (L12110249-16) and 54MW12 (L12110249-18) were reported using this continuing calibration.

- For explosives continuing calibration performed on 11/14/12 @23:31 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 02/15/12 @20:05 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No confirmation samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 11/16/12 @09:57 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation 54MW3 (L12110249-11RE) and 54MW12 (L12110249-18RE) samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 11/16/12 @18:22 on instrument HPLC4, all criteria were met. No qualifiers were applied. No confirmation samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample
Laboratory control samples (LCS) are used to monitor long-term accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used.

- Sample WG414169-03 was used as the aqueous LCS for the explosives analysis on 11/14/12. All criteria were met. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this LCS.

- Sample WG414169-03 was used as the aqueous LCS for the explosives analysis on 11/16/12. All criteria were met. Confirmation samples 54MW3 (L12110249-11RE) and 54MW12 (L12110249-18RE) apply to this LCS.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Sample 54MW1 (L12110249-01) was used as the aqueous MS/MSD for the explosives analysis on 11/14/12. All criteria were met. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54MW3 (L12110249-11RE), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), 54MW12 (L12110249-18), and 54MW12 (L12110249-18RE) apply to this MS/MSD.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for explosives analysis in this SDG; therefore, was not evaluated.

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).
- For sample 54MW3 (L12110249-11), target compound 4-amino-2,6-dinitrotoluene (56.9%) was outside confirmation criteria; therefore, were qualified estimated based upon the high RPD.
- For sample 54MW12 (L12110249-18), target compound 4-amino-2,6-dinitrotoluene (78.8%) was outside confirmation criteria; therefore, were qualified estimated based upon the high RPD.
- All other positive detections met confirmation criteria for all detected target analytes in this SDG.

Sample: 54MW3 (L12110249-11), 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.26899837
b = -0.1564446
Y = Area = 13.97858

X = 52.55

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (52.55 * 10 * 1) / (950) = 0.553 µg/L (Signal #1)

Reported Value = 0.553 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

Form I Copy

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-01
Client ID: 54MW1
Matrix: Water
Workgroup #: WG414238
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 11/14/2012 08:00
Cal Date: 02/23/2012 15:43
Run Date: 11/14/2012 13:48
File ID: 5L008648.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.05	0.263
2,4-Dinitrotoluene	121-14-2		U	1.05	0.263
2,6-Dinitrotoluene	606-20-2		U	1.05	0.263
2,4,6-Trinitrotoluene	118-96-7		U	1.05	0.263
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.05	0.263
2-Nitrotoluene	88-72-2		U	1.05	0.263
4-Nitrotoluene	99-99-0		U	1.05	0.263
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.05	0.263
RDX	121-82-4		U	1.05	0.263
Nitroglycerin	55-63-0		U	1.05	0.263

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	85.9	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	86.4	50	150	

Sample #: L12110249-07
Client ID: 54MW5
Matrix: Water
Workgroup #: WG414238
Collect Date: 11/08/2012 09:50
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 11/14/2012 08:00
Cal Date: 02/23/2012 15:43
Run Date: 11/14/2012 15:45
File ID: 5L008651.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.06	0.266
2,4-Dinitrotoluene	121-14-2		U	1.06	0.266
2,6-Dinitrotoluene	606-20-2		U	1.06	0.266
2,4,6-Trinitrotoluene	118-96-7		U	1.06	0.266
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.06	0.266
2-Nitrotoluene	88-72-2		U	1.06	0.266
4-Nitrotoluene	99-99-0		U	1.06	0.266
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.06	0.266
RDX	121-82-4		U	1.06	0.266
Nitroglycerin	55-63-0		U	1.06	0.266

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	102	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-09
Client ID: 54MW14
Matrix: Water
Workgroup #: WG414238
Collect Date: 11/08/2012 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 11/14/2012 08:00
Cal Date: 02/23/2012 15:43
Run Date: 11/14/2012 16:24
File ID: 5L008652.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.05	0.263
2,4-Dinitrotoluene	121-14-2		U	1.05	0.263
2,6-Dinitrotoluene	606-20-2		U	1.05	0.263
2,4,6-Trinitrotoluene	118-96-7		U	1.05	0.263
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.05	0.263
2-Nitrotoluene	88-72-2		U	1.05	0.263
4-Nitrotoluene	99-99-0		U	1.05	0.263
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.05	0.263
RDX	121-82-4		U	1.05	0.263
Nitroglycerin	55-63-0		U	1.05	0.263

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	90.9	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-11
Client ID: 54MW3
Matrix: Water
Workgroup #: WG414238
Collect Date: 11/08/2012 12:15
Sample Tag: CF01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC4
Prep Date: 11/14/2012 08:00
Cal Date: 02/15/2012 19:12
Run Date: 11/16/2012 13:46
File ID: 4L024458.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.05	0.263
2,4-Dinitrotoluene	121-14-2		U	1.05	0.263
2,6-Dinitrotoluene	606-20-2		U	1.05	0.263
2,4,6-Trinitrotoluene	118-96-7		U	1.05	0.263
2-Amino-4,6-dinitrotoluene	35572-78-2	0.307	J J	1.05	0.263
2-Nitrotoluene	88-72-2		U	1.05	0.263
4-Nitrotoluene	99-99-0		U	1.05	0.263
4-Amino-2,6-dinitrotoluene	19406-51-0	0.308	J J	1.05	0.263
RDX	121-82-4		U	1.05	0.263
Nitroglycerin	55-63-0		U	1.05	0.263

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	99.6	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-11	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW3	**Prep Method:**	3535	**Prep Date:**	11/14/2012 08:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG414238	**Analyst:**	ECL	**Run Date:**	11/14/2012 17:03
Collect Date:	11/08/2012 12:15	**Dilution:**	1	**File ID:**	5L008653.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.05	0.263
2,4-Dinitrotoluene	121-14-2		U	1.05	0.263
2,6-Dinitrotoluene	606-20-2		U	1.05	0.263
2,4,6-Trinitrotoluene	118-96-7		U	1.05	0.263
2-Amino-4,6-dinitrotoluene	35572-78-2	0.339	J J	1.05	0.263
2-Nitrotoluene	88-72-2		U	1.05	0.263
4-Nitrotoluene	99-99-0		U	1.05	0.263
4-Amino-2,6-dinitrotoluene	19406-51-0	0.553	J J	1.05	0.263
RDX	121-82-4		U	1.05	0.263
Nitroglycerin	55-63-0		U	1.05	0.263

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	101	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-14
Client ID: 54RB110812
Matrix: Water
Workgroup #: WG414238
Collect Date: 11/08/2012 12:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 11/14/2012 08:00
Cal Date: 02/23/2012 15:43
Run Date: 11/14/2012 17:42
File ID: 5L008654.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7		U	1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.04	0.260
RDX	121-82-4		U	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	90.3	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-16
Client ID: 54MW2
Matrix: Water
Workgroup #: WG414238
Collect Date: 11/08/2012 13:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 11/14/2012 08:00
Cal Date: 02/23/2012 15:43
Run Date: 11/14/2012 20:36
File ID: 5L008659.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7		U	1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.04	0.260
RDX	121-82-4		U	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.0	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-18	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW12	**Prep Method:**	3535	**Prep Date:**	11/14/2012 08:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG414238	**Analyst:**	ECL	**Run Date:**	11/16/2012 15:33
Collect Date:	11/08/2012 14:55	**Dilution:**	1	**File ID:**	4L024460.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7	7.47		1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2	3.27		1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0	1.50	J	1.04	0.260
RDX	121-82-4	5.16		1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	83.7	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-18	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW12	**Prep Method:**	3535	**Prep Date:**	11/14/2012 08:00
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG414238	**Analyst:**	ECL	**Run Date:**	11/14/2012 21:15
Collect Date:	11/08/2012 14:55	**Dilution:**	1	**File ID:**	5L008660.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7	7.62		1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2	3.35		1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0	3.45	J	1.04	0.260
RDX	121-82-4	6.59		1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	79.4	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L12110249

DATE: June 7, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 8, 2012. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of seven aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW1	L12110249-01	54RB110812	L12110249-14
54MW5	L12110249-07	54MW2	L12110249-16
54MW14	L12110249-09	54MW12	L12110249-18
54MW3	L12110249-11		

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
X		Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

.

EJ Malarek

Eric Malarek, Chemist

6/7/13

Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L12110249

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/08/12, the coolers were received on 11/09/12 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C, 0.0°C, and 1.0°C. Even though two of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: Samples were collected on 11/08/12. The samples were prepped and analyzed on 11/09/12 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 04/26/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
11/09/12	Chloride	ICB/CCBs	<LOD	NA	None
11/09/12	Sulfate	ICB/CCBs	<LOD	NA	None
11/09/12	Nitrate	ICB/CCBs	<LOD	NA	None
11/09/12	Chloride	WG414060-01	<½MRL	NA	None
11/09/12	Sulfate	WG414060-01	<½MRL	NA	None
11/09/12	Nitrate	WG414060-01	<½MRL	NA	None
11/09/12	Chloride	54RB110812	<½MRL	NA	None
11/09/12	Sulfate	54RB110812	<½MRL	NA	None
11/09/12	Nitrate	54RB110812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG414060-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 11/09/12. All criteria were met. No qualifiers were applied. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- Sample 54MW1 (L12110249-01) was used as the aqueous MS/MSD for the chloride, sulfate, and nitrate analysis on 11/09/12. Sulfate (111%) was above laboratory criteria. For all other anions, all criteria were met. Sulfate was qualified estimated "K" for detections and no qualification for non-detections based upon the high recovery for all associated samples. Samples 54MW1 (L12110249-01), 54MW5 (L12110249-07), 54MW14 (L12110249-09), 54MW3 (L12110249-11), 54RB110812 (L12110249-14), 54MW2 (L12110249-16), and 54MW12 (L12110249-18) apply to this MS/MSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for chloride, sulfate, and nitrate analysis in this SDG; therefore, was not evaluated.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW1 (L12110249-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1266
b = 0.00
Y = Area = 3.961
DF = 1

X = 31.29 mg/L * DF = 31.29 mg/L * 1 = 31.3 mg/L

Reported concentration = 31.3 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-01	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW1	**Prep Method:**	9056	**Prep Date:**	11/09/2012 14:31
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG414060	**Analyst:**	JBK	**Run Date:**	11/09/2012 16:32
Collect Date:	11/08/2012 07:45	**Dilution:**	1	**File ID:**	I1_110912-10
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.58		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	31.3	K	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-03
Client ID: 54MW1
Matrix: Water
Workgroup #: WG414060
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 11/09/2012 14:31
Cal Date: 04/26/2012 14:46
Run Date: 11/09/2012 17:07
File ID: I1_110912-12

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	9.46		0.200	0.100
Nitrate	14797-55-8	5.47		0.600	0.100
Sulfate	14808-79-8	75.2		1.00	0.500

Sample #: L12110249-05
Client ID: 54MW1
Matrix: Water
Workgroup #: WG414060
Collect Date: 11/08/2012 07:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 11/09/2012 14:31
Cal Date: 04/26/2012 14:46
Run Date: 11/09/2012 17:25
File ID: I1_110912-13

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	9.48		0.200	0.100
Nitrate	14797-55-8	5.48		0.600	0.100
Sulfate	14808-79-8	75.6		1.00	0.500

Sample #: L12110249-07
Client ID: 54MW5
Matrix: Water
Workgroup #: WG414060
Collect Date: 11/08/2012 09:50
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 11/09/2012 14:31
Cal Date: 04/26/2012 14:46
Run Date: 11/09/2012 17:42
File ID: I1_110912-14

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.69		0.200	0.100
Nitrate	14797-55-8	0.944		0.600	0.100
Sulfate	14808-79-8	21.5	K	1.00	0.500

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-09
Client ID: 54MW14
Matrix: Water
Workgroup #: WG414060
Collect Date: 11/08/2012 11:05
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 11/09/2012 14:31
Cal Date: 04/26/2012 14:46
Run Date: 11/09/2012 18:34
File ID: I1_110912-17

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.53		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	39.0	K	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110249-11
Client ID: 54MW3
Matrix: Water
Workgroup #: WG414060
Collect Date: 11/08/2012 12:15
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 11/09/2012 14:31
Cal Date: 04/26/2012 14:46
Run Date: 11/09/2012 18:52
File ID: I1_110912-18

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.01		0.200	0.100
Nitrate	14797-55-8	0.656		0.600	0.100
Sulfate	14808-79-8	22.9	K	1.00	0.500

Sample #: L12110249-14
Client ID: 54RB110812
Matrix: Water
Workgroup #: WG414060
Collect Date: 11/08/2012 12:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 11/09/2012 14:31
Cal Date: 04/26/2012 14:46
Run Date: 11/09/2012 19:09
File ID: I1_110912-19

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6		U	0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8		U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-16	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW2	**Prep Method:**	9056	**Prep Date:**	11/09/2012 14:31
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG414060	**Analyst:**	JBK	**Run Date:**	11/09/2012 19:26
Collect Date:	11/08/2012 13:45	**Dilution:**	1	**File ID:**	I1_110912-20
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.52		0.200	0.100
Nitrate	14797-55-8	0.327	J J	0.600	0.100
Sulfate	14808-79-8	35.4	K	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L12110249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110249-18	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW12	**Prep Method:**	9056	**Prep Date:**	11/09/2012 14:31
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG414060	**Analyst:**	JBK	**Run Date:**	11/09/2012 19:44
Collect Date:	11/08/2012 14:55	**Dilution:**	1	**File ID:**	I1_110912-21
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.40		0.200	0.100
Nitrate	14797-55-8	2.86		0.600	0.100
Sulfate	14808-79-8	49.8	K	1.00	0.500

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L12110203

DATE: May 31, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 7, 2012. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of twelve aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW13	L12110203-01	54MW11	L12110203-07
54MW13	L12110203-02	54MW11	L12110203-08
54MW9	L12110203-03	54MW10	L12110203-09
54MW9	L12110203-04	54MW10	L12110203-10
54MW8	L12110203-05	54TM10	L12110203-11
54MW8	L12110203-06	54TM10	L12110203-12

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
X		Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/31/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L12110203

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/07/12, the coolers were received on 11/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 1.0°C, 3.0°C, and 2.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The samples were collected on 11/07/12. The TOC and TIC analysis were run on 11/12/12 and 11/13/12. The DOC and DIC analysis were run on 11/12/12 and 11/13/12. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC: 1 - blank
5 - standards ($r \geq 0.995$)
ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 11/12/12 and 11/13/12. The DOC and DIC analysis were run on 11/12/12 and 11/13/12. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW13 (L12110203-01), 54MW13 (L12110203-02), 54MW9 (L12110203-03), 54MW9 (L12110203-04), 54MW8 (L12110203-05), 54MW8 (L12110203-06), 54MW11 (L12110203-07), 54MW11 (L12110203-08), 54MW10 (L12110203-09), 54MW10 (L12110203-10), 54TM10 (L12110203-11), and 54TM10 (L12110203-12) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-15) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB110812 (L12110249-14) applies to all total samples (TOC and TIC) in this SDG. The decontamination water blank TOC CK (L12110249-13) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
11/12/12	TOC	ICB/CCBs	<LOD	NA	None
11/12/12	TOC	WG413955-01	<½MRL	NA	None
11/13/12	DOC	WG413963-01	<½MRL	NA	None
11/12/12	TOC	54RB110812	0.675J	3.38	None
11/12/12	TIC	54RB110812	<½MRL	NA	None
11/13/12	DOC	54RB110812	1.62	8.10	54MW9, 54MW8, 54MW11, 54MW10, 54TM10
11/13/12	DIC	54RB110812	<½MRL	NA	None
11/12/12	TOC	TOC CK	0.778J	3.89	54TM10
11/12/12	TIC	TOC CK	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG413955-02 and WG413955-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 11/12/12. All criteria were met. No qualifiers were applied. Samples 54MW13 (L12110203-01), 54MW9 (L12110203-03), 54MW8 (L12110203-05), 54MW11 (L12110203-07), 54MW10 (L12110203-09), and 54TM10 (L12110203-11) apply to this LCS/LCSD.

- Samples WG413963-02 and WG413963-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 11/12/12. All criteria were met. No qualifiers were applied. Samples 54MW13 (L12110203-02), 54MW9 (L12110203-04), 54MW8 (L12110203-06), 54MW11 (L12110203-08), 54MW10 (L12110203-10), and 54TM10 (L12110203-12) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate
Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- No project aqueous MS/MSD was performed for TOC, TIC, DOC, and DIC analysis dated 11/12-13/12; therefore was not evaluated. See LCS Section IV for accuracy statement.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW10 (L12110203-09) and 54TM10 (L12110203-11) was collected for TOC and TIC analysis. TOC was detected at 5.55 mg/L in the parent sample and at 3.44 mg/L in the duplicate pair resulting in a RPD of 46.9% (outside limits). TIC was detected at 38.4 mg/L in the parent sample and at 37.3 mg/L in the duplicate pair resulting in a RPD of 2.9% (within limits). All criteria were met for TIC. TOC was outside limits; therefore, was qualified estimated "J" for the duplicate pair based upon the high RPD.

- Field aqueous sample duplicate pair 54MW10 (L12110203-10) and 54TM10 (L12110203-12) was collected for DOC and DIC analysis. DOC was detected at 5.45 mg/L in the parent sample and at 7.17 mg/L in the duplicate pair resulting in a RPD of 27.3% (outside limits). DIC was detected at 72.0 mg/L in the parent sample and at 74.0 mg/L in the duplicate pair resulting in a RPD of 2.7% (within limits). All criteria were met for DIC. DOC was outside limits; therefore, was qualified estimated "J" for the duplicate pair based upon the high RPD.

VIII-Quantitation Verification and Data Review

The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW13 (L12110203-01), 54MW13 (L12110203-02), 54MW9 (L12110203-03), 54MW9 (L12110203-04), 54MW8 (L12110203-05), 54MW8 (L12110203-06), 54MW11 (L12110203-07), 54MW11 (L12110203-08), 54MW10 (L12110203-09), 54MW10 (L12110203-10), 54TM10 (L12110203-11), and 54TM10 (L12110203-12) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW13	TOC/DOC	2.0; 2.0	2.00U	9.98	NA	J, UJ
54MW13	TIC/DIC	2.0; 2.0	71.1	91.8	22.6	J
54MW9	TOC/DOC	1.0; 2.0	6.73	5.10	NA	None
54MW9	TIC/DIC	1.0; 2.0	42.3	64.7	34.6	J
54MW8	TOC/DOC	1.0; 3.0	4.90	1.78J	NA	None
54MW8	TIC/DIC	1.0; 3.0	43.1	79.4	45.7	J
54MW11	TOC/DOC	1.0; 2.0	5.88	4.47	NA	None
54MW11	TIC/DIC	1.0; 2.0	41.7	78.2	46.7	J
54MW10	TOC/DOC	1.0; 2.0	5.55	5.45	NA	None
54MW10	TIC/DIC	1.0; 2.0	38.4	72.0	46.7	J
55TM10	TOC/DOC	1.0; 2.0	3.44	7.17	52.0	J
54TM10	TIC/DIC	1.0; 2.0	37.3	74.0	49.6	J

J = Estimated value.
U = Non-detect as <MRL.
MRL = Method Reporting Limit

Sample: 54MW9 (L12110203-03), TOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 1886
DF = 1

X = (48.99 mg/L) * 1 = 48.99 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 1239
DF = 1

X = (42.26 mg/L) * 1 = 42.26 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 48.99 – 42.26 = 6.73 mg/L

Reported Value = 6.73 mg/L
% Difference = 0.3%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110203-01
Client ID: 54MW13
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/07/2012 10:50
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 08:46
File ID: TC11122012.085

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		71.1	J	2.00	1.00

Sample #: L12110203-01
Client ID: 54MW13
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/07/2012 10:50
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 08:46
File ID: TC11122012.085

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC		U UJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110203-02
Client ID: 54MW13
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/07/2012 10:50
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 20:43
File ID: TC11122012.041

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		91.8	J	2.00	1.00

Sample #: L12110203-02
Client ID: 54MW13
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/07/2012 10:50
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 20:43
File ID: TC11122012.041

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	9.98	J	2.00	1.00

Sample #: L12110203-03
Client ID: 54MW9
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/07/2012 12:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 13:31
File ID: TC11122012.010

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		42.3	J	1.00	0.500

Sample #: L12110203-03
Client ID: 54MW9
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/07/2012 12:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 13:31
File ID: TC11122012.010

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	6.73		1.00	0.500

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Certificate of Analysis

Sample #: L12110203-04 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW9 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 **Analyst:** DIH **Run Date:** 11/12/2012 20:58
Collect Date: 11/07/2012 12:10 **Dilution:** 2 **File ID:** TC11122012.042
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	5.10	B	2.00	1.00

Sample #: L12110203-04 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW9 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 **Analyst:** DIH **Run Date:** 11/12/2012 20:58
Collect Date: 11/07/2012 12:10 **Dilution:** 2 **File ID:** TC11122012.042
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		64.7	J	2.00	1.00

Sample #: L12110203-05 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW8 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 **Analyst:** DIH **Run Date:** 11/12/2012 13:47
Collect Date: 11/07/2012 13:05 **Dilution:** 1 **File ID:** TC11122012.011
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	4.90		1.00	0.500

Sample #: L12110203-05 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW8 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 **Analyst:** DIH **Run Date:** 11/12/2012 13:47
Collect Date: 11/07/2012 13:05 **Dilution:** 1 **File ID:** TC11122012.011
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		43.1	J	1.00	0.500

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110203-06
Client ID: 54MW8
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/07/2012 13:05
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 3
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 09:56
File ID: TC11122012.090

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		79.4	J	3.00	1.50

Sample #: L12110203-06
Client ID: 54MW8
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/07/2012 13:05
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 3
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 09:56
File ID: TC11122012.090

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.78	J B	3.00	1.50

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110203-07
Client ID: 54MW11
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/07/2012 14:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 14:03
File ID: TC11122012.012

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	5.88		1.00	0.500

Sample #: L12110203-07
Client ID: 54MW11
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/07/2012 14:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 14:03
File ID: TC11122012.012

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		41.7	J	1.00	0.500

Sample #: L12110203-08
Client ID: 54MW11
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/07/2012 14:30
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 21:29
File ID: TC11122012.044

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		78.2	J	2.00	1.00

Sample #: L12110203-08
Client ID: 54MW11
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/07/2012 14:30
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 21:29
File ID: TC11122012.044

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.47	B	2.00	1.00

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Certificate of Analysis

Sample #: L12110203-09 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW10 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 **Analyst:** DIH **Run Date:** 11/12/2012 14:18
Collect Date: 11/07/2012 16:10 **Dilution:** 1 **File ID:** TC11122012.013
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	5.55	J	1.00	0.500

Sample #: L12110203-09 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW10 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413955 **Analyst:** DIH **Run Date:** 11/12/2012 14:18
Collect Date: 11/07/2012 16:10 **Dilution:** 1 **File ID:** TC11122012.013
Sample Tag: 01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		38.4	J	1.00	0.500

Sample #: L12110203-10 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW10 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 **Analyst:** DIH **Run Date:** 11/12/2012 21:44
Collect Date: 11/07/2012 16:10 **Dilution:** 2 **File ID:** TC11122012.045
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	5.45	B	2.00	1.00

Sample #: L12110203-10 **PrePrep Method:** N/A **Instrument:** TOC-VWP
Client ID: 54MW10 **Prep Method:** 415.1 **Prep Date:** N/A
Matrix: Water **Analytical Method:** 415.1 **Cal Date:** 08/06/2012 08:11
Workgroup #: WG413963 **Analyst:** DIH **Run Date:** 11/12/2012 21:44
Collect Date: 11/07/2012 16:10 **Dilution:** 2 **File ID:** TC11122012.045
Sample Tag: DL01 **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		72.0	J	2.00	1.00

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-11	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG413955	**Analyst:**	DIH	**Run Date:**	11/12/2012 14:57
Collect Date:	11/07/2012 16:10	**Dilution:**	1	**File ID:**	TC11122012.016
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.44	B	1.00	0.500

Sample #:	L12110203-11	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG413955	**Analyst:**	DIH	**Run Date:**	11/12/2012 14:57
Collect Date:	11/07/2012 16:10	**Dilution:**	1	**File ID:**	TC11122012.016
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		37.3	J	1.00	0.500

Sample #:	L12110203-12	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG413963	**Analyst:**	DIH	**Run Date:**	11/12/2012 21:59
Collect Date:	11/07/2012 16:10	**Dilution:**	2	**File ID:**	TC11122012.046
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		74.0	J	2.00	1.00

Sample #:	L12110203-12	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM10	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG413963	**Analyst:**	DIH	**Run Date:**	11/12/2012 21:59
Collect Date:	11/07/2012 16:10	**Dilution:**	2	**File ID:**	TC11122012.046
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	7.17	B	2.00	1.00

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L12110203

DATE: May 31, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 7, 2012. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of six aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW13	L12110203-01	54MW11	L12110203-07
54MW9	L12110203-03	54MW10	L12110203-09
54MW8	L12110203-05	54TM10	L12110203-11

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable.

Eric Malarek, Chemist

5/31/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L12110203

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/07/12, the coolers were received on 11/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 1.0°C, 3.0°C, and 2.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 11/07/12 for perchlorate analysis. The aqueous samples were prepped on 11/14/12 and analyzed on 11/15/12. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check
LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:	1- blank (<1/2MRL DoD Perchlorate Handbook)
	5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
	ICV (≤15%D DoD Perchlorate Handbook)
	CCV/ICS (≤15%D DoD Perchlorate Handbook)
	LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 11/14/12 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW13 (L12110203-01), 54MW9 (L12110203-03), 54MW8 (L12110203-05), 54MW11 (L12110203-07), 54MW10 (L12110203-09), and 54TM10 (L12110203-11) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
11/14/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
11/15/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
11/14/12	WG414299-02	All perchlorate <½MRL	NA	NA	None
11/20/12	54RB110812	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG414299-03 was used as aqueous LCS for perchlorate analysis dated 11/14/12. All criteria were met. No qualifiers were applied. Samples 54MW13 (L12110203-01), 54MW9 (L12110203-03), 54MW8 (L12110203-05), 54MW11 (L12110203-07), 54MW10 (L12110203-09), and 54TM10 (L12110203-11) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- No project aqueous MS/MSD was performed for perchlorate analysis dated 11/14/12; therefore was not evaluated. See LCS Section VII for accuracy statement.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW10 (L12110203-09) and 54TM10 (L12110203-11) was collected for perchlorate analysis. Perchlorate was detected in the original sample at 0.344 µg/L and in the duplicate pair at 0.356 µg/L; resulting in a RPD of 3.4%. All criteria were met. No qualifiers were applied.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW13 (L12110203-01), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.51
b = 0.00551
Y = Area = 32800/333000 = 0.0985
X = 0.06158

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.06158 * 5 * 1) = 0.308 µg/L (Signal #1)
Reported Value = 0.308 µg/L
% Difference = 0.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-01	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW13	**Prep Method:**	6850	**Prep Date:**	11/14/2012 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	11/14/2012 19:48
Workgroup #:	WG414299	**Analyst:**	JWR	**Run Date:**	11/15/2012 02:25
Collect Date:	11/07/2012 10:50	**Dilution:**	1	**File ID:**	1LM.LM18627
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.308		0.200	0.100

Sample #:	L12110203-03	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW9	**Prep Method:**	6850	**Prep Date:**	11/14/2012 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	11/14/2012 19:48
Workgroup #:	WG414299	**Analyst:**	JWR	**Run Date:**	11/15/2012 02:44
Collect Date:	11/07/2012 12:10	**Dilution:**	1	**File ID:**	1LM.LM18628
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-05	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW8	**Prep Method:**	6850	**Prep Date:**	11/14/2012 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	11/14/2012 19:48
Workgroup #:	WG414299	**Analyst:**	JWR	**Run Date:**	11/15/2012 03:03
Collect Date:	11/07/2012 13:05	**Dilution:**	1	**File ID:**	1LM.LM18629
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.286		0.200	0.100

Sample #:	L12110203-07	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW11	**Prep Method:**	6850	**Prep Date:**	11/14/2012 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	11/14/2012 19:48
Workgroup #:	WG414299	**Analyst:**	JWR	**Run Date:**	11/15/2012 03:22
Collect Date:	11/07/2012 14:30	**Dilution:**	1	**File ID:**	1LM.LM18630
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110203-09
Client ID: 54MW10
Matrix: Water
Workgroup #: WG414299
Collect Date: 11/07/2012 16:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/14/2012 16:00
Cal Date: 11/14/2012 19:48
Run Date: 11/15/2012 03:41
File ID: 1LM.LM18631

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.344		0.200	0.100

Sample #: L12110203-11
Client ID: 54TM10
Matrix: Water
Workgroup #: WG414299
Collect Date: 11/07/2012 16:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/14/2012 16:00
Cal Date: 11/14/2012 19:48
Run Date: 11/15/2012 04:00
File ID: 1LM.LM18632

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.356		0.200	0.100

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L12110203

DATE: May 30, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 7, 2012. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of ten aqueous samples (includes four re-analysis samples) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW13	L12110203-01	54MW11	L12110203-07
54MW13	L12110203-01RE	54MW10	L12110203-09
54MW9	L12110203-03	54MW10	L12110203-09RE
54MW8	L12110203-05	54TM10	L12110203-11
54MW8	L12110203-05RE	54TM10	L12110203-11RE

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike/Spike Duplicate
X		Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/30/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L12110203

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/07/12, the coolers were received on 11/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 1.0°C, 3.0°C, and 2.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 11/07/12. The samples were extracted on 11/09/12 and were analyzed on 11/10/12 and 11/13/12 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
11/09/12	WG413858-01	All target explosives <½MRL	NA	NA	None
11/13/12	WG413858-01	All target explosives <½MRL	NA	NA	None
11/14/12	54RB110812	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW13 (L12110203-01), 54MW9 (L12110203-03), 54MW8 (L12110203-05), 54MW11 (L12110203-07), 54MW10 (L12110203-09), and 54TM10 (L12110203-11) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Confirmation samples 54MW13 (L12110203-01RE), 54MW8 (L12110203-05RE), 54MW10 (L12110203-09RE), and 54TM10 (L12110203-11RE) apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 11/09/12 @13:14 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 11/09/12 @22:17 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW13 (L12110203-01), 54MW9 (L12110203-03), 54MW8 (L12110203-05), 54MW11 (L12110203-07), 54MW10 (L12110203-09), and 54TM10 (L12110203-11) were reported using this continuing calibration.

- For explosives continuing calibration performed on 11/10/12 @06:24 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 02/15/12 @20:05 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No confirmation samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 11/13/12 @14:48 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation 54MW13 (L12110203-01RE), 54MW8 (L12110203-05RE), 54MW10 (L12110203-09RE), and 54TM10 (L12110203-11RE) samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 11/14/12 @00:42 on instrument HPLC4, all criteria were met. No qualifiers were applied. No confirmation samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample and Laboratory Control Sample Duplicate
Laboratory control samples (LCS) and LCSD are used to monitor long-term precision and accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS/LCSD recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Samples WG413858-02 and WG413858-03 were used as the aqueous LCS/LCSD for the explosives analysis on 11/09/12. All criteria were met. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to this LCS/LCSD.

- Samples WG413858-02 and WG413858-03 were used as the aqueous LCS/LCSD for the explosives analysis on 11/13/12. All criteria were met. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to this LCS/LCSD.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- No site specific aqueous MS/MSD was analyzed for explosives analysis in this SDG; therefore, was not evaluated. See LCS/LCSD Section VI for laboratory precision and accuracy statements.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW10 (L12110203-09) and 54TM10 (L12110203-11) was analyzed for explosives analysis in this SDG. Target compound 2,4,6-trinitrotoluene was detected in the parent sample at 1.59 µg/L and in the duplicate pair at 2.74 µg/L; resulting in a RPD of 53.1%. Target compound 2-amino-4,6-dinitrotoluene was detected in the parent sample at 0.648J µg/L and in the duplicate pair at 1.05 µg/L; resulting in a RPD of 47.4%. Target compound 4-amino-2,6-dinitrotoluene was detected in the parent sample at 1.12P µg/L and in the duplicate pair at 1.47P µg/L; resulting in a RPD of 27.0%. Target compound RDX was detected in the parent sample at 1.35 µg/L and in the duplicate pair at 2.21 µg/L; resulting in a RPD of 48.3%. All other target explosives were non-detect for the duplicate pair. Target compound 2,4,6-trinitrotoluene was outside criteria; therefore, was qualified estimated "J" for the duplicate pair. For all other target compounds, all criteria were met.

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).
- For sample 54MW4 (L12110202-01), target compound RDX (70.1%) was outside confirmation criteria; therefore, were qualified estimated based upon the high RPD.
- For sample 54MW10 (L12110203-09), target compound 4-amino-2,6-dinitrotoluene (56.4%) was outside confirmation criteria; therefore, were qualified estimated based upon the high RPD.
- For sample 54TM10 (L12110203-11), target compound 4-amino-2,6-dinitrotoluene (46.9%) was outside confirmation criteria; therefore, were qualified estimated based upon the high RPD.
- All other positive detections met confirmation criteria for all detected target analytes in this SDG.

Sample: 54MW4 (L12110202-01), 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.26899837
b = -0.1564446
Y = Area = 12.96986

X = 48.8

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (48.8 * 10 * 1) / (880) = 0.555 µg/L (Signal #1)

Reported Value = 0.555 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW13	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/13/2012 19:14
Collect Date:	11/07/2012 10:50	**Dilution:**	1	**File ID:**	4L024428.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7	0.839	J J	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.10	0.275
RDX	121-82-4	0.411	J J	1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	102	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW13	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/10/2012 00:53
Collect Date:	11/07/2012 10:50	**Dilution:**	1	**File ID:**	5L008633.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7	0.843	J J	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.10	0.275
RDX	121-82-4	0.855	J J	1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	104	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110203-03
Client ID: 54MW9
Matrix: Water
Workgroup #: WG413903
Collect Date: 11/07/2012 12:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: METHOD
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 11/09/2012 08:30
Cal Date: 02/23/2012 15:43
Run Date: 11/10/2012 01:32
File ID: 5L008634.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.16	0.291
2,4-Dinitrotoluene	121-14-2		U	1.16	0.291
2,6-Dinitrotoluene	606-20-2		U	1.16	0.291
2,4,6-Trinitrotoluene	118-96-7		U	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.16	0.291
2-Nitrotoluene	88-72-2		U	1.16	0.291
4-Nitrotoluene	99-99-0		U	1.16	0.291
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.16	0.291
RDX	121-82-4		U	1.16	0.291
Nitroglycerin	55-63-0		U	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	93.0	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Sample #:	L12110203-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW8	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/10/2012 02:11
Collect Date:	11/07/2012 13:05	**Dilution:**	1	**File ID:**	5L008635.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0	0.555	J J	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	92.7	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW8	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/13/2012 20:07
Collect Date:	11/07/2012 13:05	**Dilution:**	1	**File ID:**	4L024429.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.14	0.284
2,4-Dinitrotoluene	121-14-2		U	1.14	0.284
2,6-Dinitrotoluene	606-20-2		U	1.14	0.284
2,4,6-Trinitrotoluene	118-96-7		U	1.14	0.284
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.14	0.284
2-Nitrotoluene	88-72-2		U	1.14	0.284
4-Nitrotoluene	99-99-0		U	1.14	0.284
4-Amino-2,6-dinitrotoluene	19406-51-0	0.462	J 'J'	1.14	0.284
RDX	121-82-4		U	1.14	0.284
Nitroglycerin	55-63-0		U	1.14	0.284

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	92.4	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110203-07
Client ID: 54MW11
Matrix: Water
Workgroup #: WG413903
Collect Date: 11/07/2012 14:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: METHOD
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 11/09/2012 08:30
Cal Date: 02/23/2012 15:43
Run Date: 11/10/2012 02:50
File ID: 5L008636.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	94.4	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW10	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/13/2012 21:00
Collect Date:	11/07/2012 16:10	**Dilution:**	1	**File ID:**	4L024430.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7	1.56	J	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2	0.643	J J	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0	0.627	J J	1.10	0.275
RDX	121-82-4	1.12		1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	106	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW10	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/10/2012 03:29
Collect Date:	11/07/2012 16:10	**Dilution:**	1	**File ID:**	5L008637.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7	1.59	J	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2	0.648	J	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0	1.12	J	1.10	0.275
RDX	121-82-4	1.35		1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	110	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-11	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54TM10	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/10/2012 04:08
Collect Date:	11/07/2012 16:10	**Dilution:**	1	**File ID:**	5L008638.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7	2.74	J	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2	1.05	J	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0	1.47	J	1.11	0.278
RDX	121-82-4	2.21		1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	113	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-11	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54TM10	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/13/2012 21:53
Collect Date:	11/07/2012 16:10	**Dilution:**	1	**File ID:**	4L024431.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7	2.73	J	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2	0.980	J	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0	0.912	J	1.11	0.278
RDX	121-82-4	1.74		1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	105	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Eric.malarek@CBI.com

MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L12110203

DATE: May 28, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 7, 2012. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of six aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW13	L12110203-01	54MW11	L12110203-07
54MW9	L12110203-03	54MW10	L12110203-09
54MW8	L12110203-05	54TM10	L12110203-11

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

.

E. Malarek

Eric Malarek, Chemist

5/31/13

Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L12110203

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/07/12, the coolers were received on 11/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 1.0°C, 3.0°C, and 2.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: Samples were collected on 11/07/12. The samples were prepped and analyzed on 11/08/12 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 04/26/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW13 (L12110203-01), 54MW9 (L12110203-03), 54MW8 (L12110203-05), 54MW11 (L12110203-07), 54MW10 (L12110203-09), and 54TM10 (L12110203-11) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
11/08/12	Chloride	ICB/CCBs	<LOD	NA	None
11/08/12	Sulfate	ICB/CCBs	<LOD	NA	None
11/08/12	Nitrate	ICB/CCBs	<LOD	NA	None
11/08/12	Chloride	WG413820-01	<½MRL	NA	None
11/08/12	Sulfate	WG413820-01	<½MRL	NA	None
11/08/12	Nitrate	WG413820-01	<½MRL	NA	None
11/09/12	Chloride	54RB110812	<½MRL	NA	None
11/09/12	Sulfate	54RB110812	<½MRL	NA	None
11/09/12	Nitrate	54RB110812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG413820-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 11/08/12. All criteria were met. No qualifiers were applied. Samples 54MW13 (L12110203-01), 54MW9 (L12110203-03), 54MW8 (L12110203-05), 54MW11 (L12110203-07), 54MW10 (L12110203-09), and 54TM10 (L12110203-11) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- No project aqueous MS/MSD was performed for chloride, sulfate, and nitrate analysis dated 11/08/12; therefore was not evaluated. See LCS Section IV for accuracy statement.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW10 (L12110203-09) and 54TM10 (L12110203-11) was collected for chloride, sulfate, and nitrate analysis. Chloride was detected in the parent sample at 3.71 mg/L and in the duplicate pair at 3.56 mg/L; resulting in a RPD of 4.1%. Sulfate was detected in the parent sample at 151 mg/L and in the duplicate pair at 165 mg/L; resulting in a RPD of 8.9%. Nitrate was detected at trace levels in the parent sample at 0.381J mg/L and not detected in the duplicate pair at <1.80 mg/L. No qualifiers were applied based upon this outlier. All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW13 (L12110203-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1266
b = 0.00
Y = Area = 9.704
DF = 1

X = 76.65 mg/L * DF = 76.65 mg/L * 1 = 76.7 mg/L

Reported concentration = 76.7 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-01	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW13	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 16:48
Collect Date:	11/07/2012 10:50	**Dilution:**	1	**File ID:**	I1_110812-12
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.72		0.200	0.100
Nitrate	14797-55-8	0.369	J J	0.600	0.100
Sulfate	14808-79-8	76.7		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-03	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW9	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 17:22
Collect Date:	11/07/2012 12:10	**Dilution:**	3	**File ID:**	I1_110812-14
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	2.51		0.600	0.300
Nitrate	14797-55-8		U	1.80	0.300
Sulfate	14808-79-8	199		3.00	1.50

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-05	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW8	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 18:15
Collect Date:	11/07/2012 13:05	**Dilution:**	1	**File ID:**	I1_110812-17
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.34		0.200	0.100
Nitrate	14797-55-8	0.403	J J	0.600	0.100
Sulfate	14808-79-8	60.3		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-07	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW11	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 19:24
Collect Date:	11/07/2012 14:30	**Dilution:**	1	**File ID:**	I1_110812-21
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.43		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	99.1		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-09	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW10	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 19:59
Collect Date:	11/07/2012 16:10	**Dilution:**	3	**File ID:**	I1_110812-23
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.71		0.600	0.300
Nitrate	14797-55-8	0.381	J J	1.80	0.300
Sulfate	14808-79-8	151		3.00	1.50

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12110203
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110203-11	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54TM10	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 20:16
Collect Date:	11/07/2012 16:10	**Dilution:**	3	**File ID:**	I1_110812-24
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.56		0.600	0.300
Nitrate	14797-55-8		U	1.80	0.300
Sulfate	14808-79-8	165		3.00	1.50

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L12110202

DATE: May 29, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 6, 2012. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of six aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW4	L12110202-01	54MW7	L12110202-04
54MW4	L12110202-02	54TM7	L12110202-05
54MW7	L12110202-03	54TM7	L12110202-06

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
X		Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/29/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L12110202

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/06/12, the coolers were received on 11/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 1.0°C, 3.0°C, and 2.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The samples were collected on 11/06/12. The TOC and TIC analysis were run on 11/12/12. The DOC and DIC analysis were run on 11/12/12 and 11/13/12. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC:	1 - blank
	5 - standards ($r \geq 0.995$)
	ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 11/12/12. The DOC and DIC analysis were run on 11/12/12 and 11/13/12. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW4 (L12110202-01), 54MW4 (L12110202-02), 54MW7 (L12110202-03), 54MW7 (L12110202-04), 54TM7 (L12110202-05), and 54TM7 (L12110202-06) apply to these initial and continuing calibrations.

III-Blank Analysis
The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-15) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB110812 (L12110249-14) applies to all total samples (TOC and TIC) in this SDG. The decontamination water blank TOC CK (L12110249-13) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
11/12/12	TOC	ICB/CCBs	<LOD	NA	None
11/12/12	TOC	WG413955-01	<½MRL	NA	None
11/13/12	DOC	WG413963-01	<½MRL	NA	None
11/12/12	TOC	54RB110812	0.675J	3.38	54MW4, 54TM7
11/12/12	TIC	54RB110812	<½MRL	NA	None
11/13/12	DOC	54RB110812	1.62	8.10	54MW4, 54MW7, 54TM7
11/13/12	DIC	54RB110812	<½MRL	NA	None
11/12/12	TOC	TOC CK	0.778J	3.89	54MW4, 54MW7, 54TM7
11/12/12	TIC	TOC CK	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG413955-02 and WG413955-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 11/12/12. All criteria were met. No qualifiers were applied. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to this LCS/LCSD.

- Samples WG413963-02 and WG413963-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 11/12/12. All criteria were met. No qualifiers were applied. Samples 54MW4 (L12110202-02), 54MW7 (L12110202-04), and 54TM7 (L12110202-06) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- No project aqueous MS/MSD was performed for TOC, TIC, DOC, and DIC analysis dated 11/12-13/12; therefore was not evaluated. See LCS Section IV for accuracy statement.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW7 (L12110202-03) and 54TM7 (L12110202-05) was collected for TOC and TIC analysis. TOC was detected at 3.72 mg/L in the parent sample and at 3.14 mg/L in the duplicate pair resulting in a RPD of 16.9% (within limits). TIC was detected at 43.3 mg/L in the parent sample and at 40.7 mg/L in the duplicate pair resulting in a RPD of 6.2% (within limits). All criteria were met. No qualifiers were applied.

- Field aqueous sample duplicate pair 54MW7 (L12110202-04) and 54TM7 (L12110202-06) was collected for DOC and DIC analysis. DOC was detected at 4.43 mg/L in the parent sample and at 6.42 mg/L in the duplicate pair resulting in a RPD of 36.7% (outside limits). DIC was detected at 61.8 mg/L in the parent sample and at 64.0 mg/L in the duplicate pair resulting in a RPD of 3.5% (within limits). All criteria were met for DIC. DOC was outside limits; therefore, was qualified estimated "J" for the duplicate pair based upon the high RPD.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW4 (L12110202-01), 54MW4 (L12110202-02), 54MW7 (L12110202-03), 54MW7 (L12110202-04), 54TM7 (L12110202-05), and 54TM7 (L12110202-06) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW4	TOC/DOC	1.0; 2.0	2.93	3.36	12.8	J
54MW4	TIC/DIC	1.0; 2.0	31.0	55.3	43.9	J
54MW7	TOC/DOC	1.0; 2.0	3.72	4.43	16.0	J
54MW7	TIC/DIC	1.0; 2.0	43.3	61.8	29.9	J
54TM12	TOC/DOC	1.0; 2.0	3.14	6.42	51.1	J
54TM12	TIC/DIC	1.0; 2.0	40.7	64.0	36.4	J

Sample: 54MW4 (L12110202-01), TOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 1306
DF = 1

X = (33.90 mg/L) * 1 = 33.90 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 910
DF = 1

X = (30.98 mg/L) * 1 = 30.98 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 33.90 – 30.98 = 2.92 mg/L

Reported Value = 2.93 mg/L
% Difference = 0.3%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110202-01
Client ID: 54MW4
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/06/2012 10:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 12:28
File ID: TC11122012.006

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		31.0	J	1.00	0.500

Sample #: L12110202-01
Client ID: 54MW4
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/06/2012 10:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 12:28
File ID: TC11122012.006

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.93	B	1.00	0.500

Sample #: L12110202-02
Client ID: 54MW4
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/06/2012 10:30
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 09:42
File ID: TC11122012.089

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.36	B	2.00	1.00

Sample #: L12110202-02
Client ID: 54MW4
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/06/2012 10:30
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/13/2012 09:42
File ID: TC11122012.089

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		55.3	J	2.00	1.00

Microbac

Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110202-03
Client ID: 54MW7
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/06/2012 12:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 12:43
File ID: TC11122012.007

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.72	B	1.00	0.500

Sample #: L12110202-03
Client ID: 54MW7
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/06/2012 12:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 12:43
File ID: TC11122012.007

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		43.3	J	1.00	0.500

Sample #: L12110202-04
Client ID: 54MW7
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/06/2012 12:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 19:49
File ID: TC11122012.037

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		61.8	J	2.00	1.00

Sample #: L12110202-04
Client ID: 54MW7
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/06/2012 12:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 19:49
File ID: TC11122012.037

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.43	B	2.00	1.00

Form I Copy

Microbac

Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110202-05
Client ID: 54TM7
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/06/2012 12:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 12:59
File ID: TC11122012.008

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.14	B	1.00	0.500

Sample #: L12110202-05
Client ID: 54TM7
Matrix: Water
Workgroup #: WG413955
Collect Date: 11/06/2012 12:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 12:59
File ID: TC11122012.008

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		40.7	J	1.00	0.500

Sample #: L12110202-06
Client ID: 54TM7
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/06/2012 12:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 20:27
File ID: TC11122012.040

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		64.0	J	2.00	1.00

Sample #: L12110202-06
Client ID: 54TM7
Matrix: Water
Workgroup #: WG413963
Collect Date: 11/06/2012 12:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 11/12/2012 20:27
File ID: TC11122012.040

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	6.42	B	2.00	1.00

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L12110202

DATE: May 28, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 6, 2012. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of three aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW4	L12110202-01	54TM7	L12110202-05
54MW7	L12110202-03		

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/28/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L12110202

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/06/12, the coolers were received on 11/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 1.0°C, 3.0°C, and 2.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 11/06/12 for perchlorate analysis. The aqueous samples were prepped on 11/14/12 and analyzed on 11/15/12. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check

LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:	1- blank (<1/2MRL DoD Perchlorate Handbook)
	5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
	ICV (≤15%D DoD Perchlorate Handbook)
	CCV/ICS (≤15%D DoD Perchlorate Handbook)
	LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 11/14/12 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
11/14/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
11/15/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
11/14/12	WG414299-02	All perchlorate <½MRL	NA	NA	None
11/20/12	54RB110812	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG414299-03 was used as aqueous LCS for perchlorate analysis dated 11/14/12. All criteria were met. No qualifiers were applied. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- No project aqueous MS/MSD was performed for perchlorate analysis dated 11/14/12; therefore was not evaluated. See LCS Section VII for accuracy statement.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW7 (L12110202-03) and 54TM7 (L12110202-05) was collected for perchlorate analysis. Perchlorate was detected at trace levels in the original sample at 0.103J µg/L and in the duplicate pair at 0.100J µg/L; resulting in a RPD of 3.0%. All criteria were met. No qualifiers were applied.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW4 (L12110202-01), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.51
b = 0.00551
Y = Area = 17200/358000 = 0.04804
X = 0.02817

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.02817 * 5 * 1) = 0.141 µg/L (Signal #1)
Reported Value = 0.141 µg/L
% Difference = 0.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110202-01
Client ID: 54MW4
Matrix: Water
Workgroup #: WG414299
Collect Date: 11/06/2012 10:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 11/14/2012 16:00
Cal Date: 11/14/2012 19:48
Run Date: 11/15/2012 01:29
File ID: 1LM.LM18624

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.141	J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110202-03	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW7	**Prep Method:**	6850	**Prep Date:**	11/14/2012 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	11/14/2012 19:48
Workgroup #:	WG414299	**Analyst:**	JWR	**Run Date:**	11/15/2012 01:48
Collect Date:	11/06/2012 12:00	**Dilution:**	1	**File ID:**	1LM.LM18625
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.103	J J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110202-05	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54TM7	**Prep Method:**	6850	**Prep Date:**	11/14/2012 16:00
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	11/14/2012 19:48
Workgroup #:	WG414299	**Analyst:**	JWR	**Run Date:**	11/15/2012 02:06
Collect Date:	11/06/2012 12:00	**Dilution:**	1	**File ID:**	1LM.LM18626
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.100	J J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L12110202

DATE: May 28, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 6, 2012. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of three aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW4	L12110202-01	54TM7	L12110202-05
54MW7	L12110202-03		

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable.

Eric Malarek, Chemist

5/28/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L12110202

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/06/12, the coolers were received on 11/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 1.0°C, 3.0°C, and 2.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 11/06/12. The samples were extracted on 11/09/12 and were analyzed on 11/09/12 and 11/10/12 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
11/09/12	WG413858-01	All target explosives <½MRL	NA	NA	None
11/14/12	54RB110812	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. All samples were non-detect for all target explosive analytes. No confirmation samples were required.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 11/09/12 @13:14 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 11/09/12 @22:17 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) were reported using this continuing calibration.

- For explosives continuing calibration performed on 11/10/12 @06:24 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample and Laboratory Control Sample Duplicate
Laboratory control samples (LCS) and LCSD are used to monitor long-term precision and accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS/LCSD recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Samples WG413858-02 and WG413858-03 were used as the aqueous LCS/LCSD for the explosives analysis on 11/09/12. All criteria were met. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to this LCS/LCSD.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- No site specific aqueous MS/MSD was analyzed for explosives analysis in this SDG; therefore, was not evaluated. See LCS/LCSD Section VI for laboratory precision and accuracy statements.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW7 (L12110202-03) and 54TM7 (L12110202-05) was analyzed for explosives analysis in this SDG. All target explosives were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

IX-Quantitation Verification and Data Review
The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- All samples in this SDG were non-detect for all target explosives. No further confirmations were required in this SDG.

Sample: WG413858-02, 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.26899837
b = -0.1564446
Y = Area = 143.83887

X = 535.3

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (535.3 * 10 * 1) / (1000) = 5.35 µg/L (Signal #1)

Reported Value = 5.35 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Microbac

Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12110202-01
Client ID: 54MW4
Matrix: Water
Workgroup #: WG413903
Collect Date: 11/06/2012 10:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: METHOD
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 11/09/2012 08:30
Cal Date: 02/23/2012 15:43
Run Date: 11/09/2012 22:56
File ID: 5L008630.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.09	0.272
2,4-Dinitrotoluene	121-14-2		U	1.09	0.272
2,6-Dinitrotoluene	606-20-2		U	1.09	0.272
2,4,6-Trinitrotoluene	118-96-7		U	1.09	0.272
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.09	0.272
2-Nitrotoluene	88-72-2		U	1.09	0.272
4-Nitrotoluene	99-99-0		U	1.09	0.272
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.09	0.272
RDX	121-82-4		U	1.09	0.272
Nitroglycerin	55-63-0		U	1.09	0.272

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	101	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110202-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW7	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/09/2012 23:35
Collect Date:	11/06/2012 12:00	**Dilution:**	1	**File ID:**	5L008631.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	100	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110202-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54TM7	**Prep Method:**	METHOD	**Prep Date:**	11/09/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG413903	**Analyst:**	ECL	**Run Date:**	11/10/2012 00:14
Collect Date:	11/06/2012 12:00	**Dilution:**	1	**File ID:**	5L008632.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	110	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L12110202

DATE: May 28, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on November 6, 2012. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of five aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW4	L12110202-01	54TM7	L12110202-05
54MW7	L12110202-03		

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/20/13
Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L12110202

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 11/06/12, the coolers were received on 11/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 1.0°C, 3.0°C, and 2.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: Samples were collected on 11/06/12. The samples were prepped and analyzed on 11/08/12 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 04/26/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB110812 (L12110249-14) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
11/08/12	Chloride	ICB/CCBs	<LOD	NA	None
11/08/12	Sulfate	ICB/CCBs	<LOD	NA	None
11/08/12	Nitrate	ICB/CCBs	<LOD	NA	None
11/08/12	Chloride	WG413820-01	<½MRL	NA	None
11/08/12	Sulfate	WG413820-01	<½MRL	NA	None
11/08/12	Nitrate	WG413820-01	<½MRL	NA	None
11/09/12	Chloride	54RB110812	<½MRL	NA	None
11/09/12	Sulfate	54RB110812	<½MRL	NA	None
11/09/12	Nitrate	54RB110812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG413820-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 11/08/12. All criteria were met. No qualifiers were applied. Samples 54MW4 (L12110202-01), 54MW7 (L12110202-03), and 54TM7 (L12110202-05) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- No project aqueous MS/MSD was performed for chloride, sulfate, and nitrate analysis dated 11/08/12; therefore was not evaluated. See LCS Section IV for accuracy statement.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW7 (L12110202-03) and 54TM7 (L12110202-05) was collected for chloride, sulfate, and nitrate analysis. Chloride was detected in the parent sample at 3.06 mg/L and in the duplicate pair at 3.13 mg/L; resulting in a RPD of 2.3%. Sulfate was detected in the parent sample at 179 mg/L and in the duplicate pair at 199 mg/L; resulting in a RPD of 10.6%. Nitrate was non-detect for the field duplicate pair. All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW4 (L12110202-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1266
b = 0.00
Y = Area = 10.200
DF = 5

X = 80.57 mg/L * DF = 80.57 mg/L * 5 = 403 mg/L

Reported concentration = 403 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110202-01	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW4	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 15:55
Collect Date:	11/06/2012 10:30	**Dilution:**	5	**File ID:**	I1_110812-09
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	0.825	J J	1.00	0.500
Nitrate	14797-55-8		U	3.00	0.500
Sulfate	14808-79-8	403		5.00	2.50

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110202-03	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54MW7	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 16:30
Collect Date:	11/06/2012 12:00	**Dilution:**	3	**File ID:**	I1_110812-11
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.06		0.600	0.300
Nitrate	14797-55-8		U	1.80	0.300
Sulfate	14808-79-8	179		3.00	1.50

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12110202
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12110202-05	**PrePrep Method:**	N/A	**Instrument:**	IC1
Client ID:	54TM7	**Prep Method:**	9056	**Prep Date:**	11/08/2012 14:11
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	04/26/2012 14:46
Workgroup #:	WG413820	**Analyst:**	JBK	**Run Date:**	11/08/2012 16:13
Collect Date:	11/06/2012 12:00	**Dilution:**	3	**File ID:**	I1_110812-10
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.13		0.600	0.300
Nitrate	14797-55-8		U	1.80	0.300
Sulfate	14808-79-8	199		3.00	1.50

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L12080318

DATE: May 21, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 9, 2012. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of ten aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW12	L12080318-01	54MW03	L12080318-06
54MW12	L12080318-02	54MW05	L12080318-07
54TM12	L12080318-03	54MW05	L12080318-08
54TM12	L12080318-04	54MW02	L12080318-09
54MW03	L12080318-05	54MW02	L12080318-10

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/21/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L12080318

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/09/12, the coolers were received on 08/10/12 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 0.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The samples were collected on 08/09/12. The TOC and TIC analysis were run on 08/15/12. The DOC and DIC analysis were run on 08/14/12. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC:	1 - blank
	5 - standards ($r \geq 0.995$)
	ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 08/15/12. The DOC and DIC analysis were run on 08/14/12. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW12 (L12080318-01), 54MW12 (L12080318-02), 54TM12 (L12080318-03), 54TM12 (L12080318-04), 54MW03 (L12080318-05), 54MW03 (L12080318-06), 54MW05 (L12080318-07), 54MW05 (L12080318-08), 54MW02 (L12080318-09), and 54MW02 (L12080318-10) apply to these initial and continuing calibrations.

III-Blank Analysis
The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank - 54RB080812 (L12080279-06) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB080812 (L12080279-05) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
08/14/12	TOC	ICB/CCBs	<LOD	NA	None
08/14/12	TOC	WG406302-01	<½MRL	NA	None
08/14/12	DOC	WG406302-01	<½MRL	NA	None
08/13/12	TOC	54RB080812	<½MRL	NA	None
08/13/12	TIC	54RB080812	<½MRL	NA	None
08/13/12	DOC	54RB080812	<½MRL	NA	None
08/13/12	DIC	54RB080812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG406302-02 and WG406302-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 08/14/12. All criteria were met. No qualifiers were applied. Samples 54MW12 (L12080318-01), 54TM12 (L12080318-03), 54MW03 (L12080318-05), 54MW05 (L12080318-07), and 54MW02 (L12080318-09) apply to this LCS/LCSD.

- Samples WG406302-02 and WG406302-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 08/14/12. All criteria were met. No qualifiers were applied. Samples 54MW12 (L12080318-02), 54TM12 (L12080318-04), 54MW03 (L12080318-06), 54MW05 (L12080318-08), and 54MW02 (L12080318-10) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- No project aqueous MS/MSD was performed for TOC, TIC, DOC, and DIC analysis dated 08/14/12; therefore was not evaluated. See LCS Section IV for accuracy statement.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW12 (L12080318-01) and 54TM12 (L12080318-03) was collected for TOC and TIC analysis. TOC was non-detect for the duplicate pair. TIC was detected at 1.36J mg/L in the parent sample and at 1.31J mg/L in the duplicate pair resulting in a RPD of 3.8% (within limits). All criteria were met. No qualifiers were applied.

- Field aqueous sample duplicate pair 54MW12 (L12080318-02) and 54TM12 (L12080318-04) was collected for DOC and DIC analysis. DOC was detected at 2.47 mg/L in the parent sample and at 2.74 mg/L in the duplicate pair resulting in a RPD of 10.4% (within limits). DIC was detected at 72.3 mg/L in the parent sample and at 66.8 mg/L in the duplicate pair resulting in a RPD of 7.9% (within limits). All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW12 (L12080318-01), 54MW12 (L12080318-02), 54TM12 (L12080318-03), 54TM12 (L12080318-04), 54MW03 (L12080318-05), 54MW03 (L12080318-06), 54MW05 (L12080318-07), 54MW05 (L12080318-08), 54MW02 (L12080318-09), and 54MW02 (L12080318-10) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW12	TOC/DOC	2.0; 2.0	2.00U	2.69	NA	J, UJ
54MW12	TIC/DIC	2.0; 2.0	1.39J	73.5	98.1	J
55TM12	TOC/DOC	2.0; 2.0	2.00U	2.74	NA	J, UJ
54TM12	TIC/DIC	2.0; 2.0	1.31J	66.8	98.0	J
54MW03	TOC/DOC	2.0; 2.0	2.00U	2.65	NA	J, UJ
54MW03	TIC/DIC	2.0; 2.0	1.17J	65.3	98.2	J
54MW05	TOC/DOC	2.0; 2.0	2.00U	4.03	NA	J, UJ
54MW05	TIC/DIC	2.0; 2.0	1.00J	68.1	98.5	J
54MW02	TOC/DOC	2.0; 2.0	2.00U	2.47	NA	J, UJ
54MW02	TIC/DIC	2.0; 2.0	1.36J	72.3	98.1	J

J = Estimated value.
U = Value non-detect as <MRL.
MRL = Method Reporting Limit
NA = Not Applicable

Sample: 54MW12 (L12080318-02), DOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 1468
DF = 1

X = (38.12 mg/L) * 2 = 76.24 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 1079
DF = 1

X = (36.77 mg/L) * 2 = 73.54 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 76.24 – 73.54 = 2.70 mg/L

Reported Value = 2.69 mg/L
% Difference = 0.4%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW12	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406302	**Analyst:**	JBK	**Run Date:**	08/15/2012 05:45
Collect Date:	08/09/2012 08:00	**Dilution:**	2	**File ID:**	TC08152012.060
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		1.39	J	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW12	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406302	**Analyst:**	JBK	**Run Date:**	08/15/2012 05:45
Collect Date:	08/09/2012 08:00	**Dilution:**	2	**File ID:**	TC08152012.060
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U VJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-02
Client ID: 54MW12
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 14:06
File ID: TC08152012.012

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		73.5	J	2.00	1.00

Sample #: L12080318-02
Client ID: 54MW12
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 14:06
File ID: TC08152012.012

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		2.69	J	2.00	1.00

Sample #: L12080318-03
Client ID: 54TM12
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/15/2012 05:57
File ID: TC08152012.061

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U UJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-03	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM12	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406302	**Analyst:**	JBK	**Run Date:**	08/15/2012 05:57
Collect Date:	08/09/2012 08:00	**Dilution:**	2	**File ID:**	TC08152012.061
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		1.31	J J	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-04
Client ID: 54TM12
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 14:20
File ID: TC08152012.013

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		66.8	J	2.00	1.00

Sample #: L12080318-04
Client ID: 54TM12
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 14:20
File ID: TC08152012.013

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		2.74	J	2.00	1.00

Sample #: L12080318-05
Client ID: 54MW03
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 09:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/15/2012 06:08
File ID: TC08152012.062

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U UJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-05	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW03	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406302	**Analyst:**	JBK	**Run Date:**	08/15/2012 06:08
Collect Date:	08/09/2012 09:00	**Dilution:**	2	**File ID:**	TC08152012.062
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		1.17	J J	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-06
Client ID: 54MW03
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 09:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 14:56
File ID: TC08152012.016

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		65.3	J	2.00	1.00

Sample #: L12080318-06
Client ID: 54MW03
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 09:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 14:56
File ID: TC08152012.016

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		2.65	J	2.00	1.00

Sample #: L12080318-07
Client ID: 54MW05
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 10:20
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/15/2012 06:20
File ID: TC08152012.063

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		1.00	J J	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW05	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406302	**Analyst:**	JBK	**Run Date:**	08/15/2012 06:20
Collect Date:	08/09/2012 10:20	**Dilution:**	2	**File ID:**	TC08152012.063
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U UJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-08
Client ID: 54MW05
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 10:20
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 15:11
File ID: TC08152012.017

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		68.1	J	2.00	1.00

Sample #: L12080318-08
Client ID: 54MW05
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 10:20
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 15:11
File ID: TC08152012.017

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		4.03	J	2.00	1.00

Sample #: L12080318-09
Client ID: 54MW02
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 12:10
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/15/2012 06:35
File ID: TC08152012.064

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		1.36	J J	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-09	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW02	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406302	**Analyst:**	JBK	**Run Date:**	08/15/2012 06:35
Collect Date:	08/09/2012 12:10	**Dilution:**	2	**File ID:**	TC08152012.064
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U UJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-10
Client ID: 54MW02
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 12:10
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 15:25
File ID: TC08152012.018

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		2.47	J	2.00	1.00

Sample #: L12080318-10
Client ID: 54MW02
Matrix: Water
Workgroup #: WG406302
Collect Date: 08/09/2012 12:10
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: JBK
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 15:25
File ID: TC08152012.018

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		72.3	J	2.00	1.00

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L12080318

DATE: May 21, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 9, 2012. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of five aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW12	L12080318-01	54MW05	L12080318-07
54TM12	L12080318-03	54MW02	L12080318-09
54MW03	L12080318-05		

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable.

Eric Malarek, Chemist

5/21/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L12080318

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/09/12, the coolers were received on 08/10/12 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 0.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 08/09/12 for perchlorate analysis. The aqueous samples were prepped on 08/16/12 and analyzed on 08/17/12. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check

LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:
1- blank (<1/2MRL DoD Perchlorate Handbook)
5 – standards (r≥0.995 or RSD≤20% DoD Perchlorate Handbook)
ICV (≤15%D DoD Perchlorate Handbook)
CCV/ICS (≤15%D DoD Perchlorate Handbook)
LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 07/23/12 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW12 (L12080318-01), 54TM12 (L12080318-03), 54MW03 (L12080318-05), 54MW05 (L12080318-07), and 54MW02 (L12080318-09) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
08/16/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
08/16/12	WG406595-02	All perchlorate <½MRL	NA	NA	None
08/17/12	54RB080812	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG406595-03 was used as aqueous LCS for perchlorate analysis dated 08/16/12. All criteria were met. No qualifiers were applied. Samples 54MW12 (L12080318-01), 54TM12 (L12080318-03), 54MW03 (L12080318-05), 54MW05 (L12080318-07), and 54MW02 (L12080318-09) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- No project aqueous MS/MSD was performed for perchlorate analysis dated 08/16/12; therefore was not evaluated. See LCS Section VII for accuracy statement.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW12 (L12080318-01) and 54TM12 (L12080318-03) was collected for perchlorate analysis. Perchlorate was detected in the original sample at 2.00 µg/L and in the duplicate pair at 2.36 µg/L; resulting in a RPD of 16.5%. All criteria were met. No qualifiers were applied.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW12 (L12080318-01), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.26
b = 0.000333
Y = Area = 40300/399000 = 0.101
X = 0.079896

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.079896 * 5 * 5) = 2.00 µg/L (Signal #1)
Reported Value = 2.00 µg/L
% Difference = 0.0%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

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Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-01
Client ID: 54MW12
Matrix: Water
Workgroup #: WG406595
Collect Date: 08/09/2012 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 5
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 17:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 00:36
File ID: 1LM.LM17328

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	2.00		1.00	0.500

Sample #: L12080318-03
Client ID: 54TM12
Matrix: Water
Workgroup #: WG406595
Collect Date: 08/09/2012 08:00
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 5
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 17:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 00:55
File ID: 1LM.LM17329

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	2.36		1.00	0.500

Sample #: L12080318-05
Client ID: 54MW03
Matrix: Water
Workgroup #: WG406595
Collect Date: 08/09/2012 09:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 17:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 01:14
File ID: 1LM.LM17330

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.405		0.200	0.100

Sample #: L12080318-07
Client ID: 54MW05
Matrix: Water
Workgroup #: WG406595
Collect Date: 08/09/2012 10:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 17:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 01:33
File ID: 1LM.LM17331

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.420		0.200	0.100

Form I Copy

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-09	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW02	**Prep Method:**	6850	**Prep Date:**	08/16/2012 17:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406595	**Analyst:**	JWR	**Run Date:**	08/17/2012 01:51
Collect Date:	08/09/2012 12:10	**Dilution:**	1	**File ID:**	1LM.LM17332
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L12080318

DATE: May 20, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 9, 2012. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of nine aqueous samples (includes four confirmation re-analysis sample) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW12	L12080318-01	54MW03	L12080318-05RE
54MW12	L12080318-01RE	54MW05	L12080318-07
54TM12	L12080318-03	54MW05	L12080318-07RE
54TM12	L12080318-03RE	54MW02	L12080318-09
54MW03	L12080318-05		

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/20/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L12080318

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/09/12, the coolers were received on 08/10/12 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 0.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: The aqueous samples were collected on 08/09/12. The samples were extracted on 08/14/12 and were analyzed on 08/15/12 and 08/16/12 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
08/15/12	WG406276-02	All target explosives <½MRL	NA	NA	None
08/16/12	WG406276-02	All target explosives <½MRL	NA	NA	None
08/15/12	54RB080812	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW12 (L12080318-01), 54TM12 (L12080318-03), 54MW03 (L12080318-05), 54MW05 (L12080318-07), and 54MW02 (L12080318-09) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Confirmation samples 54MW12 (L12080318-01RE), 54TM12 (L12080318-03RE), 54MW03 (L12080318-05RE), and 54MW05 (L12080318-07RE) apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 08/15/12 @10:34 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/15/12 @19:35 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW12 (L12080318-01), 54TM12 (L12080318-03), 54MW03 (L12080318-05), 54MW05 (L12080318-07), and 54MW02 (L12080318-09) were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/16/12 @03:03 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 02/15/12 @20:05 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No confirmation samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 08/16/12 @15:59 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation samples 54MW12 (L12080318-01RE), 54TM12 (L12080318-03RE), 54MW03 (L12080318-05RE), and 54MW05 (L12080318-07RE) were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/17/12 @01:54 on instrument HPLC4, all criteria were met. No qualifiers were applied. No confirmation samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample
Laboratory control samples (LCS) are used to monitor long-term accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used.

- Sample WG406276-03 was used as the aqueous LCS for the explosives analysis on 08/15/12. All criteria were met. Samples 54MW12 (L12080318-01), 54TM12 (L12080318-03), 54MW03 (L12080318-05), 54MW05 (L12080318-07), and 54MW02 (L12080318-09) apply to this LCS.

- Sample WG406276-03 was used as the aqueous LCS for the explosives analysis on 08/16/12. All criteria were met. Confirmation samples 54MW12 (L12080318-01RE), 54TM12 (L12080318-03RE), 54MW03 (L12080318-05RE), and 54MW05 (L12080318-07RE) applies to this LCS.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Sample 54MW04 (L12080279-09) was used as the aqueous MS/MSD for the explosives analysis on 08/15/12. Target compounds 2-amino-4,6-dinitrotoluene (RPD=20.3%) and 4-amino-2,6-dinitrotoluene (20.4%) were outside precision criteria. All recoveries were within criteria for all target explosives. The spiked sample was non-detect for all target explosives; therefore, no qualifiers were applied based upon the high RPDs. Samples 54MW12 (L12080318-01), 54MW12 (L12080318-01RE), 54TM12 (L12080318-03), 54TM12 (L12080318-03RE), 54MW03 (L12080318-05), 54MW03 (L12080318-05RE), 54MW05 (L12080318-07), 54MW05 (L12080318-07RE), 54MW02 (L12080318-09) apply to this MS/MSD.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW12 (L12080318-01) and 54TM12 (L12080318-03) was analyzed for explosives analysis in this SDG. Target compound 2,4,6-Trinitrotoluene was detected in the parent sample at 10.1 µg/L and in the duplicate pair at 10.3 µg/L; resulting in a RPD of 2.0%. Target compound 2-amino-4,6-dinitrotoluene was detected in the parent sample at 5.32 µg/L and in the duplicate pair at 5.30 µg/L; resulting in a RPD of 0.4%. Target compound 4-amino-2,6-dinitrotoluene was detected in the parent sample at 7.69 µg/L and in the duplicate pair at 7.71 µg/L; resulting in a RPD of 0.3%. Target compound RDX was detected in the parent sample at 1.95 µg/L and in the duplicate pair at 1.95 µg/L; resulting in a RPD of 0.0%. All other target explosives were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

IX-Quantitation Verification and Data Review
The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- For sample 54MW12 (L12080318-01), target compounds 4-amino-2,6-dinitrotoluene (91%) and RDX (118%) were outside confirmation criteria; therefore, were qualified estimated based upon the high RPDs.

- For sample 54TM12 (L12080318-03), target compounds 4-amino-2,6-dinitrotoluene (84%) and RDX (116%) were outside confirmation criteria; therefore, were qualified estimated based upon the high RPDs.

- All other positive detections met confirmation criteria for all detected target analytes in this SDG.

Sample: 54MW12 (L12080318-01), 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.26899837
b = -0.1564446
Y = Area = 192.26808

X = 715.34

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (715.34 * 10 * 1) / (930) = 7.69 µg/L (Signal #1)

Reported Value = 7.69 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

Form I Copy

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-01
Client ID: 54MW12
Matrix: Water
Workgroup #: WG406370
Collect Date: 08/09/2012 08:00
Sample Tag: CF01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC4
Prep Date: 08/14/2012 08:30
Cal Date: 02/15/2012 19:12
Run Date: 08/16/2012 20:25
File ID: 4L024277.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.08	0.269
2,4-Dinitrotoluene	121-14-2		U	1.08	0.269
2,6-Dinitrotoluene	606-20-2		U	1.08	0.269
2,4,6-Trinitrotoluene	118-96-7	10.0		1.08	0.269
2-Amino-4,6-dinitrotoluene	35572-78-2	5.32		1.08	0.269
2-Nitrotoluene	88-72-2		U	1.08	0.269
4-Nitrotoluene	99-99-0		U	1.08	0.269
4-Amino-2,6-dinitrotoluene	19406-51-0	2.87	J	1.08	0.269
RDX	121-82-4	0.499	J J	1.08	0.269
Nitroglycerin	55-63-0		U	1.08	0.269

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	98.7	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-01
Client ID: 54MW12
Matrix: Water
Workgroup #: WG406370
Collect Date: 08/09/2012 08:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 08/14/2012 08:30
Cal Date: 02/23/2012 15:43
Run Date: 08/15/2012 21:32
File ID: 5L008207.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.08	0.269
2,4-Dinitrotoluene	121-14-2		U	1.08	0.269
2,6-Dinitrotoluene	606-20-2		U	1.08	0.269
2,4,6-Trinitrotoluene	118-96-7	10.1		1.08	0.269
2-Amino-4,6-dinitrotoluene	35572-78-2	5.32		1.08	0.269
2-Nitrotoluene	88-72-2		U	1.08	0.269
4-Nitrotoluene	99-99-0		U	1.08	0.269
4-Amino-2,6-dinitrotoluene	19406-51-0	7.69	J	1.08	0.269
RDX	121-82-4	1.95	J	1.08	0.269
Nitroglycerin	55-63-0		U	1.08	0.269

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	90.1	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-03
Client ID: 54TM12
Matrix: Water
Workgroup #: WG406370
Collect Date: 08/09/2012 08:00
Sample Tag: CF01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC4
Prep Date: 08/14/2012 08:30
Cal Date: 02/15/2012 19:12
Run Date: 08/16/2012 21:18
File ID: 4L024278.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7	10.2		1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2	5.47		1.04	0.260
2-Nitrotoluene	88-72-2	1.21		1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0	3.16	J	1.04	0.260
RDX	121-82-4	0.513	J	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	101	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-03
Client ID: 54TM12
Matrix: Water
Workgroup #: WG406370
Collect Date: 08/09/2012 08:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 08/14/2012 08:30
Cal Date: 02/23/2012 15:43
Run Date: 08/15/2012 22:11
File ID: 5L008208.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7	10.3		1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2	5.30		1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0	7.71	J	1.04	0.260
RDX	121-82-4	1.95	J	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	82.0	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW03	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/16/2012 22:11
Collect Date:	08/09/2012 09:00	**Dilution:**	1	**File ID:**	4L024279.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2	0.393	J J	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0	0.390	J J	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.7	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW03	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/15/2012 22:50
Collect Date:	08/09/2012 09:00	**Dilution:**	1	**File ID:**	5L008209.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2	0.425	J J	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0	0.466	J J	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.8	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-07	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW05	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/16/2012 23:04
Collect Date:	08/09/2012 10:20	**Dilution:**	1	**File ID:**	4L024280.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.02	0.255
2,4-Dinitrotoluene	121-14-2		U	1.02	0.255
2,6-Dinitrotoluene	606-20-2		U	1.02	0.255
2,4,6-Trinitrotoluene	118-96-7		U	1.02	0.255
2-Amino-4,6-dinitrotoluene	35572-78-2	0.296	J J	1.02	0.255
2-Nitrotoluene	88-72-2		U	1.02	0.255
4-Nitrotoluene	99-99-0		U	1.02	0.255
4-Amino-2,6-dinitrotoluene	19406-51-0	0.301	J J	1.02	0.255
RDX	121-82-4		U	1.02	0.255
Nitroglycerin	55-63-0		U	1.02	0.255

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.1	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-07
Client ID: 54MW05
Matrix: Water
Workgroup #: WG406370
Collect Date: 08/09/2012 10:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 08/14/2012 08:30
Cal Date: 02/23/2012 15:43
Run Date: 08/15/2012 23:30
File ID: 5L008210.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.02	0.255
2,4-Dinitrotoluene	121-14-2		U	1.02	0.255
2,6-Dinitrotoluene	606-20-2		U	1.02	0.255
2,4,6-Trinitrotoluene	118-96-7		U	1.02	0.255
2-Amino-4,6-dinitrotoluene	35572-78-2	0.318	J J	1.02	0.255
2-Nitrotoluene	88-72-2		U	1.02	0.255
4-Nitrotoluene	99-99-0		U	1.02	0.255
4-Amino-2,6-dinitrotoluene	19406-51-0	0.360	J J	1.02	0.255
RDX	121-82-4		U	1.02	0.255
Nitroglycerin	55-63-0		U	1.02	0.255

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	96.7	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080318-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW02	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/16/2012 00:09
Collect Date:	08/09/2012 12:10	**Dilution:**	1	**File ID:**	5L008211.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.12	0.281
2,4-Dinitrotoluene	121-14-2		U	1.12	0.281
2,6-Dinitrotoluene	606-20-2		U	1.12	0.281
2,4,6-Trinitrotoluene	118-96-7		U	1.12	0.281
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.12	0.281
2-Nitrotoluene	88-72-2		U	1.12	0.281
4-Nitrotoluene	99-99-0		U	1.12	0.281
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.12	0.281
RDX	121-82-4		U	1.12	0.281
Nitroglycerin	55-63-0		U	1.12	0.281

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.4	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Eric.malarek@CBI.com

MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L12080318

DATE: May 21, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 9, 2012. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of five aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW12	L12080318-01	54MW05	L12080318-07
54TM12	L12080318-03	54MW02	L12080318-09
54MW03	L12080318-05		

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/21/13
Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L12080318

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/09/12, the coolers were received on 08/10/12 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 0.0°C. Even though one of the coolers were received below criteria, there were no impacts to data quality and no qualifiers were applied based upon this outlier.

- Holding Time Review: Samples were collected on 08/09/12. The samples were prepped and analyzed on 08/10/12 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 04/26/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW12 (L12080318-01), 54TM12 (L12080318-03), 54MW03 (L12080318-05), 54MW05 (L12080318-07), and 54MW02 (L12080318-09) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
08/10/12	Chloride	ICB/CCBs	<LOD	NA	None
08/10/12	Sulfate	ICB/CCBs	<LOD	NA	None
08/10/12	Nitrate	ICB/CCBs	<LOD	NA	None
08/10/12	Chloride	WG406300-01	<½MRL	NA	None
08/10/12	Sulfate	WG406300-01	<½MRL	NA	None
08/10/12	Nitrate	WG406300-01	<½MRL	NA	None
08/09/12	Chloride	54RB080812	<½MRL	NA	None
08/09/12	Sulfate	54RB080812	<½MRL	NA	None
08/09/12	Nitrate	54RB080812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG406300-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 08/10/12. All criteria were met. No qualifiers were applied. Samples 54MW12 (L12080318-01), 54TM12 (L12080318-03), 54MW03 (L12080318-05), 54MW05 (L12080318-07), and 54MW02 (L12080318-09) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- No project aqueous MS/MSD was performed for chloride, sulfate, and nitrate analysis dated 08/10/12; therefore was not evaluated. See LCS Section IV for accuracy statement.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW12 (L12080318-01) and 54TM12 (L12080318-03) was collected for chloride, sulfate, and nitrate analysis. Chloride was detected in the parent sample at 5.35 mg/L and in the duplicate pair at 5.41 mg/L; resulting in a RPD of 1.1%. Sulfate was detected in the parent sample at 27.0 mg/L and in the duplicate pair at 27.1 mg/L; resulting in a RPD of 0.4%. Nitrate was detected in the parent sample at 1.06 mg/L and in the duplicate pair at 1.05 mg/L; resulting in a RPD of 1.0%. All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW12 (L12080318-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1266
b = 0.00
Y = Area = 3.410
DF = 1

X = 26.94 mg/L * DF = 26.94 mg/L * 1 = 27.0 mg/L

Reported concentration = 27.0 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-01
Client ID: 54MW12
Matrix: Water
Workgroup #: WG406300
Collect Date: 08/09/2012 08:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 08/10/2012 15:20
Cal Date: 04/26/2012 14:46
Run Date: 08/10/2012 19:41
File ID: I10810121941.18

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.35		0.200	0.100
Nitrate	14797-55-8	1.06		0.600	0.100
Sulfate	14808-79-8	27.0		1.00	0.500

Sample #: L12080318-03
Client ID: 54TM12
Matrix: Water
Workgroup #: WG406300
Collect Date: 08/09/2012 08:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 08/10/2012 15:20
Cal Date: 04/26/2012 14:46
Run Date: 08/10/2012 19:58
File ID: I10810121958.19

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.41		0.200	0.100
Nitrate	14797-55-8	1.05		0.600	0.100
Sulfate	14808-79-8	27.1		1.00	0.500

Sample #: L12080318-05
Client ID: 54MW03
Matrix: Water
Workgroup #: WG406300
Collect Date: 08/09/2012 09:00
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 08/10/2012 15:20
Cal Date: 04/26/2012 14:46
Run Date: 08/10/2012 20:15
File ID: I10810122015.20

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.09		0.200	0.100
Nitrate	14797-55-8	0.613		0.600	0.100
Sulfate	14808-79-8	23.8		1.00	0.500

Form I Copy

Microbac

Lab Report #: L12080318
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080318-07
Client ID: 54MW05
Matrix: Water
Workgroup #: WG406300
Collect Date: 08/09/2012 10:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 08/10/2012 15:20
Cal Date: 04/26/2012 14:46
Run Date: 08/10/2012 20:33
File ID: I10810122033.21

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.87		0.200	0.100
Nitrate	14797-55-8	0.728		0.600	0.100
Sulfate	14808-79-8	18.0		1.00	0.500

Sample #: L12080318-09
Client ID: 54MW02
Matrix: Water
Workgroup #: WG406300
Collect Date: 08/09/2012 12:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 1
Units: mg/L

Instrument: IC1
Prep Date: 08/10/2012 15:20
Cal Date: 04/26/2012 14:46
Run Date: 08/10/2012 20:50
File ID: I10810122050.22

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.43		0.200	0.100
Nitrate	14797-55-8	0.190	J J	0.600	0.100
Sulfate	14808-79-8	95.5		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L12080279

DATE: May 15, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 8, 2012. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of fourteen aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW01	L12080279-01	54MW06	L12080279-08
54MW01	L12080279-02	54MW04	L12080279-09
54TM01	L12080279-03	54MW04	L12080279-10
54TM01	L12080279-04	54MW07	L12080279-15
54RB080812	L12080279-05	54MW07	L12080279-16
54RB080812	L12080279-06	54MW14	L12080279-17
54MW06	L12080279-07	54MW14	L12080279-18

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
X		Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/15/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L12080279

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/08/12, the coolers were received on 08/09/12 by the primary laboratory (Microbac Laboratories, Inc.) at 6.0°C and 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The samples were collected on 08/08/12. The TOC and TIC analysis were run on 08/13/12. The DOC and DIC analysis were run on 08/13/12 and 08/14/12. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC:	1 - blank
	5 - standards ($r \geq 0.995$)
	ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 08/13/12. The DOC and DIC analysis were run on 08/13/12 and 08/14/12. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54MW01 (L12080279-02), 54TM01 (L12080279-03), 54TM01 (L12080279-04), 54RB080812 (L12080279-05), 54RB080812 (L12080279-06), 54MW06 (L12080279-07), 54MW06 (L12080279-08), 54MW04 (L12080279-09), 54MW04 (L12080279-10), 54MW07 (L12080279-15), 54MW07 (L12080279-16), 54MW14 (L12080279-17), and 54MW14 (L12080279-18) apply to these initial and continuing calibrations.

III-Blank Analysis
The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank - 54RB080812 (L12080279-06) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB080812 (L12080279-05) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
08/13/12	TOC	ICB/CCBs	<LOD	NA	None
08/13/12	TOC	WG406212-01	<½MRL	NA	None
08/13/12	DOC	WG406214-01	<½MRL	NA	None
08/13/12	TOC	54RB080812	<½MRL	NA	None
08/13/12	TIC	54RB080812	<½MRL	NA	None
08/13/12	DOC	54RB080812	<½MRL	NA	None
08/13/12	DIC	54RB080812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG406212-02 and WG406212-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 08/13/12. All criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this LCS/LCSD.

- Samples WG406214-02 and WG406214-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 08/13/12. All criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-02), 54TM01 (L12080279-04), 54RB080812 (L12080279-06), 54MW06 (L12080279-08), 54MW04 (L12080279-10), 54MW07 (L12080279-16), and 54MW14 (L12080279-18) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- Sample 54MW04 (L12080279-09) was used as the aqueous MS/MSD for TOC and TIC analysis on 08/13/12. All criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this MS/MSD.

- Sample 54MW04 (L12080279-10) was used as the aqueous MS/MSD for DOC and DIC analysis on 08/13/12. All criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-02), 54TM01 (L12080279-04), 54RB080812 (L12080279-06), 54MW06 (L12080279-08), 54MW04 (L12080279-10), 54MW07 (L12080279-16), and 54MW14 (L12080279-18) apply to this MS/MSD.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW01 (L12080279-01) and 54TM01 (L12080279-03) was collected for TOC and TIC analysis. TOC was detected at trace levels at 0.721J mg/L in the parent sample and non-detect at <1.00 mg/L in the duplicate pair. No qualifiers were applied based upon this outlier. TIC was detected at 26.6 mg/L in the parent sample and at 24.6 mg/L in the duplicate pair resulting in a RPD of 7.8% (within limits). All criteria were met. No qualifiers were applied.

- Field aqueous sample duplicate pair 54MW01 (L12080279-02) and 54TM01 (L12080279-04) was collected for DOC and DIC analysis. DOC was detected at 1.54 mg/L in the parent sample and at 0.725J mg/L in the duplicate pair resulting in a RPD of 72.0% (outside limits). DIC was detected at 38.5 mg/L in the parent sample and at 7.57 mg/L in the duplicate pair resulting in a RPD of 134% (outside limits). DOC and DIC were qualified estimated "J" for the duplicate pair based upon these outliers.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW01 (L12080279-01), 54MW01 (L12080279-02), 54TM01 (L12080279-03), 54TM01 (L12080279-04), 54RB080812 (L12080279-05), 54RB080812 (L12080279-06), 54MW06 (L12080279-07), 54MW06 (L12080279-08), 54MW04 (L12080279-09), 54MW04 (L12080279-10), 54MW07 (L12080279-15), 54MW07 (L12080279-16), 54MW14 (L12080279-17), and 54MW14 (L12080279-18) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW01	TOC/DOC	1.0; 1.0	0.721J	1.54	53.2	J
54MW01	TIC/DIC	1.0; 1.0	26.6	38.5	30.9	J
54MW04	TOC/DOC	1.0; 1.0	1.10	2.43	54.7	J
54MW04	TIC/DIC	1.0; 1.0	27.5	44.4	38.1	J
54MW06	TOC/DOC	1.0; 1.0	1.00U	1.54	NA	J, UJ
54MW06	TIC/DIC	1.0; 1.0	35.8	34.0	NA	None
54MW07	TOC/DOC	1.0; 2.0	1.02	2.00U	NA	None
54MW07	TIC/DIC	1.0; 2.0	34.5	45.8	24.7	J
54MW14	TOC/DOC	1.0; 2.0	2.04	2.15	5.1	None
54MW14	TIC/DIC	1.0; 2.0	38.9	57.3	32.1	J
54TM01	TOC/DOC	1.0; 1.0	1.00U	0.725J	NA	None
54TM01	TIC/DIC	1.0; 1.0	24.6	7.57	NA	None
54RB080812	TOC/DOC	1.0; 1.0	1.00U	1.00U	NA	None
54RB080812	TIC/DIC	1.0; 1.0	1.00U	1.00U	NA	None

J = Estimated value.
U = Value non-detect as <MRL.
MRL = Method Reporting Limit
NA = Not Applicable

Sample: 54MW01 (L12080279-01), TOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 1054
DF = 1

X = (27.35 mg/L) * 1 = 27.35 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 783.3
DF = 1

X = (26.63 mg/L) * 1 = 26.63 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 27.35 – 26.63 = 0.720 mg/L

Reported Value = 0.721 mg/L
% Difference = 0.1%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW01	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 13:41
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	TC08132012.012
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		26.6	J	1.00	0.500

Sample #:	L12080279-01	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW01	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 13:41
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	TC08132012.012
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon		0.721	J J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080279-02
Client ID: 54MW01
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/08/2012 07:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 20:37
File ID: TC08132012.044

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		1.54	J	1.00	0.500

Sample #: L12080279-02
Client ID: 54MW01
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/08/2012 07:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 20:37
File ID: TC08132012.044

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		38.5	J	1.00	0.500

Sample #: L12080279-03
Client ID: 54TM01
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/08/2012 07:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 13:55
File ID: TC08132012.013

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		24.6		1.00	0.500

Sample #: L12080279-03
Client ID: 54TM01
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/08/2012 07:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 13:55
File ID: TC08132012.013

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-04	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM01	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 20:51
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	TC08132012.045
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		7.57	J	1.00	0.500

Sample #:	L12080279-04	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54TM01	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 20:51
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	TC08132012.045
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		0.725	J J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-05	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB080812	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 14:29
Collect Date:	08/08/2012 08:15	**Dilution:**	1	**File ID:**	TC08132012.016
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-05	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB080812	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 14:29
Collect Date:	08/08/2012 08:15	**Dilution:**	1	**File ID:**	TC08132012.016
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-06	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB080812	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 21:03
Collect Date:	08/08/2012 08:15	**Dilution:**	1	**File ID:**	TC08132012.046
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-06	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54RB080812	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 21:03
Collect Date:	08/08/2012 08:15	**Dilution:**	1	**File ID:**	TC08132012.046
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW06	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 14:42
Collect Date:	08/08/2012 12:05	**Dilution:**	1	**File ID:**	TC08132012.017
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		35.8		1.00	0.500

Sample #:	L12080279-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW06	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 14:42
Collect Date:	08/08/2012 12:05	**Dilution:**	1	**File ID:**	TC08132012.017
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U U5	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-08	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW06	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 21:15
Collect Date:	08/08/2012 12:05	**Dilution:**	1	**File ID:**	TC08132012.047
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		1.54	J	1.00	0.500

Sample #:	L12080279-08	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW06	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 21:15
Collect Date:	08/08/2012 12:05	**Dilution:**	1	**File ID:**	TC08132012.047
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		34.0		1.00	0.500

Sample #:	L12080279-09	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW04	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 14:55
Collect Date:	08/08/2012 13:30	**Dilution:**	1	**File ID:**	TC08132012.018
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		27.5	J	1.00	0.500

Sample #:	L12080279-09	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW04	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 14:55
Collect Date:	08/08/2012 13:30	**Dilution:**	1	**File ID:**	TC08132012.018
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon		1.10	J	1.00	0.500

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Certificate of Analysis

Sample #: L12080279-10
Client ID: 54MW04
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/08/2012 13:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 21:30
File ID: TC08132012.048

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		44.4	J	1.00	0.500

Sample #: L12080279-10
Client ID: 54MW04
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/08/2012 13:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 21:30
File ID: TC08132012.048

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		2.43	J	1.00	0.500

Sample #: L12080279-11
Client ID: MS8812
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/08/2012 13:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 15:09
File ID: TC08132012.019

Analyte	CAS #	Result	Qual	LOQ	LOD
~~Total Inorganic Carbon~~		~~17.4~~		~~1.00~~	~~0.500~~

Sample #: L12080279-11
Client ID: MS8812
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/08/2012 13:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 15:09
File ID: TC08132012.019

Analyte	CAS #	Result	Qual	LOQ	LOD
~~Total Organic Carbon~~		~~10.9~~		~~1.00~~	~~0.500~~

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080279-14
Client ID: MSD8812
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/08/2012 13:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 22:37
File ID: TC08132012.053

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		11.8		1.00	0.500

Sample #: L12080279-14
Client ID: MSD8812
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/08/2012 13:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 22:37
File ID: TC08132012.053

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		39.5		1.00	0.500

Sample #: L12080279-15
Client ID: 54MW07
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/08/2012 15:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 15:40
File ID: TC08132012.021

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		34.5	J	1.00	0.500

Sample #: L12080279-15
Client ID: 54MW07
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/08/2012 15:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 15:40
File ID: TC08132012.021

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon		1.02		1.00	0.500

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-16	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW07	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/14/2012 08:29
Collect Date:	08/08/2012 15:10	**Dilution:**	2	**File ID:**	TC08132012.073
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved			U	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080279-16
Client ID: 54MW07
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/08/2012 15:10
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 08:29
File ID: TC08132012.073

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		45.8	J	2.00	1.00

Sample #: L12080279-17
Client ID: 54MW14
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/08/2012 16:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 16:00
File ID: TC08132012.022

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon		2.04		1.00	0.500

Sample #: L12080279-17
Client ID: 54MW14
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/08/2012 16:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 16:00
File ID: TC08132012.022

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		38.9	J	1.00	0.500

Sample #: L12080279-18
Client ID: 54MW14
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/08/2012 16:20
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 08:42
File ID: TC08132012.074

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		2.15		2.00	1.00

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-18	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW14	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/14/2012 08:42
Collect Date:	08/08/2012 16:20	**Dilution:**	2	**File ID:**	TC08132012.074
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		57.3	J	2.00	1.00

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Fax: +1 (410) 273-7103
Eric.malarek@CBI.com

MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L12080279

DATE: May 15, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 8, 2012. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of seven aqueous samples (includes one rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW01	L12080279-01	54MW04	L12080279-09
54TM01	L12080279-03	54MW07	L12080279-15
54RB080812	L12080279-05	54MW14	L12080279-17
54MW06	L12080279-07		

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/15/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L12080279

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/08/12, the coolers were received on 08/09/12 by the primary laboratory (Microbac Laboratories, Inc.) at 6.0°C and 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 08/08/12 for perchlorate analysis. The aqueous samples were prepped on 08/16/12 and analyzed on 08/17/12. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check
LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:
1- blank (<1/2MRL DoD Perchlorate Handbook)
5 – standards ($r \geq 0.995$ or RSD≤20% DoD Perchlorate Handbook)
ICV (≤15%D DoD Perchlorate Handbook)
CCV/ICS (≤15%D DoD Perchlorate Handbook)
LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 07/23/12 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
08/17/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
08/17/12	WG406597-02	All perchlorate <½MRL	NA	NA	None
08/17/12	54RB080812	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG406597-03 was used as aqueous LCS for perchlorate analysis dated 08/17/12. All criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- Sample 54MW04 (L12080279-09) was used as aqueous MS/MSD for perchlorate analysis dated 08/17/12. All criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this MS/MSD sample.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW01 (L12080279-01) and 54TM01 (L12080279-03) was collected for perchlorate analysis. Perchlorate was non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW06 (L12080279-07), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.26
b = 0.000333
Y = Area = 16600/375000 = 0.0443
X = 0.0349

Conc. μg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in μg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (μg/L)
DF = Dilution factor

Conc. μg/L = (0.0349 * 5 * 1) = 0.174 μg/L (Signal #1)

Reported Value = 0.175 μg/L
% Difference = 0.6%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-01	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW01	**Prep Method:**	6850	**Prep Date:**	08/16/2012 19:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406597	**Analyst:**	JWR	**Run Date:**	08/17/2012 05:58
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	1LM.LM17345
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-03	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54TM01	**Prep Method:**	6850	**Prep Date:**	08/16/2012 19:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406597	**Analyst:**	JWR	**Run Date:**	08/17/2012 06:17
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	1LM.LM17346
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-05	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54RB080812	**Prep Method:**	6850	**Prep Date:**	08/16/2012 19:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406597	**Analyst:**	JWR	**Run Date:**	08/17/2012 07:32
Collect Date:	08/08/2012 08:15	**Dilution:**	1	**File ID:**	1LM.LM17350
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-07	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW06	**Prep Method:**	6850	**Prep Date:**	08/16/2012 19:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406597	**Analyst:**	JWR	**Run Date:**	08/17/2012 07:51
Collect Date:	08/08/2012 12:05	**Dilution:**	1	**File ID:**	1LM.LM17351
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.175	J	0.200	0.100

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-09	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW04	**Prep Method:**	6850	**Prep Date:**	08/16/2012 19:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406597	**Analyst:**	JWR	**Run Date:**	08/17/2012 08:10
Collect Date:	08/08/2012 13:30	**Dilution:**	1	**File ID:**	1LM.LM17352
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080279-11
Client ID: MS8812
Matrix: Water
Workgroup #: WG406597
Collect Date: 08/08/2012 13:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 19:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 08:29
File ID: 1LM.LM17353

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.218		0.200	0.100

Sample #: L12080279-13
Client ID: MSD8812
Matrix: Water
Workgroup #: WG406597
Collect Date: 08/08/2012 13:30
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 19:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 08:48
File ID: 1LM.LM17354

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.203		0.200	0.100

Sample #: L12080279-15
Client ID: 54MW07
Matrix: Water
Workgroup #: WG406597
Collect Date: 08/08/2012 15:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 19:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 09:07
File ID: 1LM.LM17355

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-17	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW14	**Prep Method:**	6850	**Prep Date:**	08/16/2012 19:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406597	**Analyst:**	JWR	**Run Date:**	08/17/2012 09:26
Collect Date:	08/08/2012 16:20	**Dilution:**	1	**File ID:**	1LM.LM17356
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.240		0.200	0.100

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L12080279

DATE: May 13, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 8, 2012. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of eight aqueous samples (includes one confirmation re-analysis sample and one rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW01	L12080279-01	54MW04	L12080279-09
54TM01	L12080279-03	54MW07	L12080279-15
54RB080812	L12080279-05	54MW14	L12080279-17
54MW06	L12080279-07	54MW14	L12080279-17RE

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/13/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L12080279

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/08/12, the coolers were received on 08/09/12 by the primary laboratory (Microbac Laboratories, Inc.) at 6.0°C and 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 08/08/12. The samples were extracted on 08/14/12 and were analyzed on 08/15/12 and 08/16/12 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
08/15/12	WG406276-02	All target explosives <½MRL	NA	NA	None
08/16/12	WG406276-02	All target explosives <½MRL	NA	NA	None
08/15/12	54RB080812	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Confirmation sample 54MW14 (L12080279-17RE) applies to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 08/15/12 @10:34 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), and 54MW04 (L12080279-09) were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/15/12 @19:35 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW07 (L12080279-15), and 54MW14 (L12080279-17) were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/16/12 @03:03 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 02/15/12 @20:05 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No confirmation samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 08/16/12 @15:59 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation sample 54MW14 (L12080279-17RE) was reported using this continuing calibration.

- For explosives continuing calibration performed on 08/17/12 @01:54 on instrument HPLC4, all criteria were met. No qualifiers were applied. No confirmation samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample
Laboratory control samples (LCS) are used to monitor long-term accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used.

- Sample WG406276-03 was used as the aqueous LCS for the explosives analysis on 08/15/12. All criteria were met. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this LCS.

- Sample WG406276-03 was used as the aqueous LCS for the explosives analysis on 08/16/12. All criteria were met. Confirmation sample 54MW14 (L12080279-17RE) applies to this LCS.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- Sample 54MW04 (L12080279-09) was used as the aqueous MS/MSD for the explosives analysis on 08/15/12. Target compounds 2-amino-4,6-dinitrotoluene (RPD=20.3%) and 4-amino-2,6-dinitrotoluene (20.4%) were outside precision criteria. All recoveries were within criteria for all target explosives. The spiked sample was non-detect for all target explosives; therefore, no qualifiers were applied based upon the high RPDs. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), 54MW14 (L12080279-17), and 54MW14 (L12080279-17RE) apply to this MS/MSD.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- Field groundwater sample duplicate pair 54MW01 (L12080279-01) and 54TM01 (L12080279-03) was analyzed for explosives analysis in this SDG. All target explosives were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- All positive detections met confirmation criteria for all detected target analytes in this SDG.

Sample: 54MW14 (L12080279-17), 4-amino-2,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.26899837
b = -0.1564446
Y = Area = 10.02863

X = 37.863

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (37.86 * 10 * 1) / (960) = 0.394 µg/L (Signal #1)

Reported Value = 0.394 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-01	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW01	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/15/2012 13:26
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	5L008194.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.02	0.255
2,4-Dinitrotoluene	121-14-2		U	1.02	0.255
2,6-Dinitrotoluene	606-20-2		U	1.02	0.255
2,4,6-Trinitrotoluene	118-96-7		U	1.02	0.255
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.02	0.255
2-Nitrotoluene	88-72-2		U	1.02	0.255
4-Nitrotoluene	99-99-0		U	1.02	0.255
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.02	0.255
RDX	121-82-4		U	1.02	0.255
Nitroglycerin	55-63-0		U	1.02	0.255

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.1	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54TM01	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/15/2012 14:05
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	5L008195.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.06	0.266
2,4-Dinitrotoluene	121-14-2		U	1.06	0.266
2,6-Dinitrotoluene	606-20-2		U	1.06	0.266
2,4,6-Trinitrotoluene	118-96-7		U	1.06	0.266
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.06	0.266
2-Nitrotoluene	88-72-2		U	1.06	0.266
4-Nitrotoluene	99-99-0		U	1.06	0.266
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.06	0.266
RDX	121-82-4		U	1.06	0.266
Nitroglycerin	55-63-0		U	1.06	0.266

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	110	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-05	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54RB080812	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/15/2012 14:44
Collect Date:	08/08/2012 08:15	**Dilution:**	1	**File ID:**	5L008196.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7		U	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.10	0.275
RDX	121-82-4		U	1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	106	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-07	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW06	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/15/2012 15:23
Collect Date:	08/08/2012 12:05	**Dilution:**	1	**File ID:**	5L008197.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.10	0.275
2,4-Dinitrotoluene	121-14-2		U	1.10	0.275
2,6-Dinitrotoluene	606-20-2		U	1.10	0.275
2,4,6-Trinitrotoluene	118-96-7		U	1.10	0.275
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.10	0.275
2-Nitrotoluene	88-72-2		U	1.10	0.275
4-Nitrotoluene	99-99-0		U	1.10	0.275
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.10	0.275
RDX	121-82-4		U	1.10	0.275
Nitroglycerin	55-63-0		U	1.10	0.275

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	97.4	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW04	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/15/2012 16:02
Collect Date:	08/08/2012 13:30	**Dilution:**	1	**File ID:**	5L008198.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.08	0.269
2,4-Dinitrotoluene	121-14-2		U	1.08	0.269
2,6-Dinitrotoluene	606-20-2		U	1.08	0.269
2,4,6-Trinitrotoluene	118-96-7		U	1.08	0.269
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.08	0.269
2-Nitrotoluene	88-72-2		U	1.08	0.269
4-Nitrotoluene	99-99-0		U	1.08	0.269
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.08	0.269
RDX	121-82-4		U	1.08	0.269
Nitroglycerin	55-63-0		U	1.08	0.269

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	96.0	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	111	50	150	

Sample #: L12080279-15
Client ID: 54MW07
Matrix: Water
Workgroup #: WG406370
Collect Date: 08/08/2012 15:10
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 08/14/2012 08:30
Cal Date: 02/23/2012 15:43
Run Date: 08/15/2012 20:14
File ID: 5L008205.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.11	0.278
2,4-Dinitrotoluene	121-14-2		U	1.11	0.278
2,6-Dinitrotoluene	606-20-2		U	1.11	0.278
2,4,6-Trinitrotoluene	118-96-7		U	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.11	0.278
RDX	121-82-4		U	1.11	0.278
Nitroglycerin	55-63-0		U	1.11	0.278

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	95.5	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-17	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW14	**Prep Method:**	3535	**Prep Date:**	08/14/2012 08:30
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG406370	**Analyst:**	ECL	**Run Date:**	08/16/2012 19:32
Collect Date:	08/08/2012 16:20	**Dilution:**	1	**File ID:**	4L024276.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7		U	1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0	0.304	J	1.04	0.260
RDX	121-82-4		U	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	107	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080279-17
Client ID: 54MW14
Matrix: Water
Workgroup #: WG406370
Collect Date: 08/08/2012 16:20
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 08/14/2012 08:30
Cal Date: 02/23/2012 15:43
Run Date: 08/15/2012 20:53
File ID: 5L008206.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7		U	1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0	0.394	J	1.04	0.260
RDX	121-82-4		U	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	104	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L12080279

DATE: May 13, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 8, 2012. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of seven aqueous samples (includes one rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW01	L12080279-01	54MW04	L12080279-09
54TM01	L12080279-03	54MW07	L12080279-15
54RB080812	L12080279-05	54MW14	L12080279-17
54MW06	L12080279-07		

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

.

Eric Malarek, Chemist

5/13/13

Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L12080279

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/08/12, the coolers were received on 08/09/12 by the primary laboratory (Microbac Laboratories, Inc.) at 6.0°C and 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: Samples were collected on 08/08/12. The samples were prepped and analyzed on 08/09/12 and 08/10/12 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 07/26/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
08/09/12	Chloride	ICB/CCBs	<LOD	NA	None
08/09/12	Sulfate	ICB/CCBs	<LOD	NA	None
08/09/12	Nitrate	ICB/CCBs	<LOD	NA	None
08/09/12	Chloride	WG406031-01	<½MRL	NA	None
08/09/12	Sulfate	WG406031-01	<½MRL	NA	None
08/09/12	Nitrate	WG406031-01	<½MRL	NA	None
08/09/12	Chloride	54RB080812	<½MRL	NA	None
08/09/12	Sulfate	54RB080812	<½MRL	NA	None
08/09/12	Nitrate	54RB080812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG406031-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 08/09/12. All criteria were met. No qualifiers were applied. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- Sample 54MW04 (L12080279-09) was used as the aqueous MS/MSD for the chloride, sulfate, and nitrate analysis on 08/09/12. Sulfate (114%) was outside criteria. All RPDs were within criteria for all target anions. The sample concentration was >4 times the spike added for sulfate; therefore, no qualifiers were applied based upon this outlier. Samples 54MW01 (L12080279-01), 54TM01 (L12080279-03), 54RB080812 (L12080279-05), 54MW06 (L12080279-07), 54MW04 (L12080279-09), 54MW07 (L12080279-15), and 54MW14 (L12080279-17) apply to this MS/MSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- Field aqueous sample duplicate pair 54MW01 (L12080279-01) and 54TM01 (L12080279-03) was collected for chloride, sulfate, and nitrate analysis. Chloride was detected in the parent sample at 1.63 mg/L and in the duplicate pair at 1.74 mg/L; resulting in a RPD of 6.5%. Sulfate was detected in the parent sample at 26.2 mg/L and in the duplicate pair at 25.8 mg/L; resulting in a RPD of 1.5%. Nitrate was non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW01 (L12080279-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1121
b = -0.0220
Y = Area = 2.912
DF = 1

X = 26.2 mg/L * DF = 31.40 mg/L * 1 = 26.2 mg/L
Reported concentration = 26.2 mg/L
%D = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-01	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW01	**Prep Method:**	9056	**Prep Date:**	08/09/2012 14:56
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG406031	**Analyst:**	JBK	**Run Date:**	08/09/2012 20:46
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	I20809122046.22
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.63		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	26.2		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-03	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54TM01	**Prep Method:**	9056	**Prep Date:**	08/09/2012 14:56
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG406031	**Analyst:**	JBK	**Run Date:**	08/09/2012 21:04
Collect Date:	08/08/2012 07:40	**Dilution:**	1	**File ID:**	I20809122104.23
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.74		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	25.8		1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-05	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54RB080812	**Prep Method:**	9056	**Prep Date:**	08/09/2012 14:56
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG406031	**Analyst:**	JBK	**Run Date:**	08/09/2012 21:23
Collect Date:	08/08/2012 08:15	**Dilution:**	1	**File ID:**	I20809122123.24
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6		U	0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8		U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-07	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW06	**Prep Method:**	9056	**Prep Date:**	08/09/2012 14:56
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG406031	**Analyst:**	JBK	**Run Date:**	08/09/2012 21:41
Collect Date:	08/08/2012 12:05	**Dilution:**	1	**File ID:**	I20809122141.25
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.04		0.200	0.100
Nitrate	14797-55-8	0.120	J J	0.600	0.100
Sulfate	14808-79-8	15.7		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-09	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW04	**Prep Method:**	9056	**Prep Date:**	08/09/2012 14:56
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG406031	**Analyst:**	JBK	**Run Date:**	08/09/2012 22:00
Collect Date:	08/08/2012 13:30	**Dilution:**	5	**File ID:**	I20809122200.26
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.36		1.00	0.500
Nitrate	14797-55-8		U	3.00	0.500
Sulfate	14808-79-8	378		5.00	2.50

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080279-11
Client ID: MS8812
Matrix: Water
Workgroup #: WG406031
Collect Date: 08/08/2012 13:30
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 5
Units: mg/L

Instrument: IC2
Prep Date: 08/09/2012 14:56
Cal Date: 07/26/2012 14:25
Run Date: 08/09/2012 23:13
File ID: I20809122313.30

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	9.28		1.00	0.500
Nitrate	14797-55-8	5.24		3.00	0.500
Sulfate	14808-79-8	423		5.00	2.50

Sample #: L12080279-13
Client ID: MSD8812
Matrix: Water
Workgroup #: WG406031
Collect Date: 08/08/2012 13:30
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 5
Units: mg/L

Instrument: IC2
Prep Date: 08/09/2012 14:56
Cal Date: 07/26/2012 14:25
Run Date: 08/09/2012 23:32
File ID: I20809122332.31

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	8.85		1.00	0.500
Nitrate	14797-55-8	5.21		3.00	0.500
Sulfate	14808-79-8	420		5.00	2.50

Sample #: L12080279-15
Client ID: 54MW07
Matrix: Water
Workgroup #: WG406031
Collect Date: 08/08/2012 15:10
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 9056
Analytical Method: 9056
Analyst: JBK
Dilution: 5
Units: mg/L

Instrument: IC2
Prep Date: 08/09/2012 14:56
Cal Date: 07/26/2012 14:25
Run Date: 08/09/2012 23:50
File ID: I20809122350.32

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	2.38		1.00	0.500
Nitrate	14797-55-8		U	3.00	0.500
Sulfate	14808-79-8	186		5.00	2.50

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080279
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080279-17	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW14	**Prep Method:**	9056	**Prep Date:**	08/09/2012 14:56
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG406031	**Analyst:**	JBK	**Run Date:**	08/10/2012 00:09
Collect Date:	08/08/2012 16:20	**Dilution:**	1	**File ID:**	I20810120009.33
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.22		0.200	0.100
Nitrate	14797-55-8	0.214	J J	0.600	0.100
Sulfate	14808-79-8	31.8		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L12080249

DATE: May 2, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 7, 2012. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of twelve aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L12080249-01	54MW08	L12080249-07
54MW09	L12080249-02	54MW08	L12080249-08
54MW11	L12080249-03	54MW10	L12080249-09
54MW11	L12080249-04	54MW10	L12080249-10
54DI8712	L12080249-05	54MW13	L12080249-11
54DI8712	L12080249-06	54MW13	L12080249-12

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/3/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L12080249

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/07/12, the cooler was received on 08/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The samples were collected on 08/07/12. The TOC and TIC analysis were run on 08/13/12 and 08/14/12. The DOC and DIC analysis were run on 08/13/12. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifier was applied.

II-Initial and Continuing Calibration
Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC:	1 - blank
	5 - standards ($r \geq 0.995$)
	ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 08/13/12 and 08/14/12. The DOC and DIC analysis were run on 08/13/12. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW09 (L12080249-01), 54MW09 (L12080249-02), 54MW11 (L12080249-03), 54MW11 (L12080249-04), 54DI8712 (L12080249-05), 54DI8712 (L12080249-06), 54MW08 (L12080249-07), 54MW08 (L12080249-08), 54MW10 (L12080249-09), 54MW10 (L12080249-10), 54MW13 (L12080249-11), and 54MW13 (L12080249-12) apply to these initial and continuing calibrations.

III-Blank Analysis
The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank - 54RB080812 (L12080279-06) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB080812 (L12080279-05) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
08/13/12	TOC	ICB/CCBs	<LOD	NA	None
08/13/12	TOC	WG406212-01	<½MRL	NA	None
08/13/12	DOC	WG406214-01	<½MRL	NA	None
08/13/12	TOC	54RB080812	<½MRL	NA	None
08/13/12	TIC	54RB080812	<½MRL	NA	None
08/13/12	DOC	54RB080812	<½MRL	NA	None
08/13/12	DIC	54RB080812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG406212-02 and WG406212-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 08/13/12. All criteria were met. No qualifiers were applied. Samples 54MW09 (L12080249-01), 54MW11 (L12080249-03), 54DI8712 (L12080249-05), 54MW08 (L12080249-07), 54MW10 (L12080249-09), and 54MW13 (L12080249-11) apply to this LCS/LCSD.

- Samples WG406214-02 and WG406214-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 08/13/12. All criteria were met. No qualifiers were applied. Samples 54MW09 (L12080249-02), 54MW11 (L12080249-04), 54DI8712 (L12080249-06), 54MW08 (L12080249-08), 54MW10 (L12080249-10), and 54MW13 (L12080249-12) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate

Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- No project specific aqueous MS/MSD was performed for the TOC, TIC, DOC, and DIC analysis for this SDG; therefore, was not evaluated. See Section IV for laboratory accuracy and precision statements using LCS/LCSD.

VI-Laboratory Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG; therefore, was not evaluated.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW09 (L12080249-01), 54MW09 (L12080249-02), 54MW11 (L12080249-03), 54MW11 (L12080249-04), 54DI8712 (L12080249-05), 54DI8712 (L12080249-06), 54MW08 (L12080249-07), 54MW08 (L12080249-08), 54MW10 (L12080249-09), 54MW10 (L12080249-10), 54MW13 (L12080249-11), and 54MW13 (L12080249-12) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW09	TOC/DOC	2.0; 2.0	2.00U	2.60	NA	J, UJ
54MW09	TIC/DIC	2.0; 2.0	6.58	67.3	90.2	J
54MW11	TOC/DOC	2.0; 2.0	2.00U	38.1	NA	J, UJ
54MW11	TIC/DIC	2.0; 2.0	5.45	29.2	81.3	J
54DI8712	TOC/DOC	1.0; 1.0	0.524J	1.00U	NA	None
54DI8712	TIC/DIC	1.0; 1.0	1.00U	1.00U	0.0	None
54MW08	TOC/DOC	2.0; 2.0	2.00U	5.54	NA	J, UJ
54MW08	TIC/DIC	2.0; 2.0	5.72	8.70	34.3	J
54MW10	TOC/DOC	2.0; 2.0	1.37J	32.7	95.8	J
54MW10	TIC/DIC	2.0; 2.0	6.73	37.7	82.2	J
54MW13	TOC/DOC	2.0; 2.0	1.05J	72.2	98.6	J
54MW13	TIC/DIC	2.0; 2.0	5.32	6.60	19.4	J

J = Estimated value.
U = Value non-detect as <MRL.
MRL = Method Reporting Limit
NA = Not Applicable

Sample: 54MW10 (L12080249-09), TOC

TC: Y = m*X (mg/L) + b	TIC: Y = m*X (mg/L) + b
m = 38.46	m = 29.16
b = 2.031	b = 6.703
Y = 157.8	Y = 104.8
DF = 2	DF = 2
X = (4.05 mg/L) * 2 = 8.10 mg/L	X = (3.36 mg/L) * 2 = 6.72 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 8.10 – 6.72 = 1.38 mg/L

Reported Value = 1.37 mg/L
% Difference = 0.7%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-01
Client ID: 54MW09
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/07/2012 10:25
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 00:13
File ID: TC08132012.060

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		6.58	J	2.00	1.00

Sample #: L12080249-01
Client ID: 54MW09
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/07/2012 10:25
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 00:13
File ID: TC08132012.060

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U UJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Certificate of Analysis

Sample #:	L12080249-02	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW09	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 18:57
Collect Date:	08/07/2012 10:25	**Dilution:**	2	**File ID:**	TC08132012.036
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		67.3	J	2.00	1.00

Sample #:	L12080249-02	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW09	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 18:57
Collect Date:	08/07/2012 10:25	**Dilution:**	2	**File ID:**	TC08132012.036
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		2.60	J	2.00	1.00

Sample #:	L12080249-03	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW11	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/14/2012 00:25
Collect Date:	08/07/2012 11:40	**Dilution:**	2	**File ID:**	TC08132012.061
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		5.45	J	2.00	1.00

Sample #:	L12080249-03	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW11	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/14/2012 00:25
Collect Date:	08/07/2012 11:40	**Dilution:**	2	**File ID:**	TC08132012.061
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U UJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-04
Client ID: 54MW11
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/07/2012 11:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 19:10
File ID: TC08132012.037

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		29.2	J	2.00	1.00

Sample #: L12080249-04
Client ID: 54MW11
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/07/2012 11:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 19:10
File ID: TC08132012.037

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		38.1	J	2.00	1.00

Sample #: L12080249-05
Client ID: 54DI8712
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/07/2012 11:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 12:42
File ID: TC08132012.008

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-05	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54DI8712	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/13/2012 12:42
Collect Date:	08/07/2012 11:40	**Dilution:**	1	**File ID:**	TC08132012.008
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon		0.524	J J	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

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Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-06	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54DI8712	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 19:44
Collect Date:	08/07/2012 11:40	**Dilution:**	1	**File ID:**	TC08132012.040
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-06	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54DI8712	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406214	**Analyst:**	DIH	**Run Date:**	08/13/2012 19:44
Collect Date:	08/07/2012 11:40	**Dilution:**	1	**File ID:**	TC08132012.040
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved			U	1.00	0.500

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW08	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/14/2012 00:59
Collect Date:	08/07/2012 12:45	**Dilution:**	2	**File ID:**	TC08132012.064
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		5.72	J	2.00	1.00

Sample #:	L12080249-07	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW08	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG406212	**Analyst:**	DIH	**Run Date:**	08/14/2012 00:59
Collect Date:	08/07/2012 12:45	**Dilution:**	2	**File ID:**	TC08132012.064
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon			U UJ	2.00	1.00

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-08
Client ID: 54MW08
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/07/2012 12:45
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 19:56
File ID: TC08132012.041

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		5.54	J	2.00	1.00

Sample #: L12080249-08
Client ID: 54MW08
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/07/2012 12:45
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 19:56
File ID: TC08132012.041

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		8.70	J	2.00	1.00

Sample #: L12080249-09
Client ID: 54MW10
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/07/2012 13:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 01:11
File ID: TC08132012.065

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon		1.37	J J	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-09
Client ID: 54MW10
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/07/2012 13:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 01:11
File ID: TC08132012.065

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		6.73	J	2.00	1.00

Sample #: L12080249-10
Client ID: 54MW10
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/07/2012 13:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 20:09
File ID: TC08132012.042

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		37.7	J	2.00	1.00

Sample #: L12080249-10
Client ID: 54MW10
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/07/2012 13:40
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 20:09
File ID: TC08132012.042

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		32.7	J	2.00	1.00

Sample #: L12080249-11
Client ID: 54MW13
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/07/2012 15:25
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 01:23
File ID: TC08132012.066

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon		1.05	J J	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

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Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-11
Client ID: 54MW13
Matrix: Water
Workgroup #: WG406212
Collect Date: 08/07/2012 15:25
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/14/2012 01:23
File ID: TC08132012.066

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		5.32	J	2.00	1.00

Sample #: L12080249-12
Client ID: 54MW13
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/07/2012 15:25
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 20:23
File ID: TC08132012.043

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved		72.2	J	2.00	1.00

Sample #: L12080249-12
Client ID: 54MW13
Matrix: Water
Workgroup #: WG406214
Collect Date: 08/07/2012 15:25
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: DIH
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 08/13/2012 20:23
File ID: TC08132012.043

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		6.60	J	2.00	1.00

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L12080249

DATE: May 2, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 7, 2012. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Microbac Laboratories. A total of five samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L12080249-01	54MW10	L12080249-09
54MW11	L12080249-03	54MW13	L12080249-11
54MW08	L12080249-07		

Data were reviewed and validated by Eric Malarek using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), *DoD Perchlorate Handbook August, Rev1, Change 1, 2007* (DoD, 2007), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Instrument Performance Check
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Internal Standards
	X	Interference Check Sample
	X	Laboratory Control Sample (LCS)
	X	Matrix Spike (MS) and Spike Duplicate (MSD)
	X	Field Duplicate
	X	Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable.

Eric Malarek, Chemist

5/2/13
Date

RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L12080249

I-Holding Times and Preservation
The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. For perchlorate analysis, aqueous samples are received and stored at cool @4°C±2°C with a maximum holding time of 28 days from collection (DoD Perchlorate Handbook criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/07/12, the cooler was received on 08/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 08/07/12 for perchlorate analysis. The aqueous samples were prepped on 08/16/12 and analyzed on 08/17/12. Sample collection dates may be found on the attached form 1s. All holding time criteria were met. No qualifiers were applied.

II-Instrument Performance Check
LC/MS instrument performance checks are performed to ensure mass resolution, identification and, to some degree, sensitivity. The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples are analyzed.

- The instrument performance check, ^{18}O-Perchlorate, met the mass calibration criteria. No qualification was applied.

III-Initial and Continuing Calibration
Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Perchlorate:	1- blank (<1/2MRL DoD Perchlorate Handbook)
	5 – standards ($r \geq 0.995$ or RSD≤20% DoD Perchlorate Handbook)
	ICV (≤15%D DoD Perchlorate Handbook)
	CCV/ICS (≤15%D DoD Perchlorate Handbook)
	LODV (±30%D DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 07/23/12 on instrument LCMS1, all criteria were met for all target compounds (RSD≤20%). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with correlation coefficients ≥0.995 for primary and confirmation columns. Samples 54MW09 (L12080249-01), 54MW11 (L12080249-03), 54MW08 (L12080249-07), 54MW10 (L12080249-09), and 54MW13 (L12080249-11) apply to this initial calibration.

IV-Blanks

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be detected in any of the associated blanks >MDL. The DoD Perchlorate Handbook criterion specifies all concentrations should be less than ½ MRL for method blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5X) the maximum amount for target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
08/17/12	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
08/17/12	WG406597-02	All perchlorate <½MRL	NA	NA	None
08/17/12	54RB080812	All perchlorate <½MRL	NA	NA	None

MRL = Method Reporting Limit.
NA = Not Applicable.

V-Internal Standards

Internal standards performance criteria ensure that LC/MS sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard. The DoD Handbook specifies retention times (RT) 1.0 ± 2% of last calibration standard and the ratio of RT of sample to standard should be 3.06 and fall between 2.3 and 3.8. The internal standard peak area responses should fall between 50% to 150% recoveries.

- All criteria were met. No qualifiers were applied.

VI-Interference Check Sample (ICS)

The LCMS interference check sample (ICS) verifies inter-element and background correction factors. LCMS Interference Check is performed at the beginning and end of each sample analysis run. Control limits are 70-130% (DoD QSM limits 70-130%).

- All criteria were met. No qualifiers were applied.

VII-Laboratory Control Sample

The laboratory control sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. DoD Perchlorate Handbook and laboratory aqueous limits are 80-120%.

- Sample WG406579-03 was used as aqueous LCS for perchlorate analysis dated 08/17/12. All criteria were met. No qualifiers were applied. Samples 54MW09 (L12080249-01), 54MW11 (L12080249-03), 54MW08 (L12080249-07), 54MW10 (L12080249-09), and 54MW13 (L12080249-11) apply to this LCS sample.

VIII-Matrix Spike (MS) and Spike Duplicate (MSD)
Matrix Spike (MS) and Spike Duplicate (MSD) are generated to determine long-term accuracy and precision of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. MS/MSD recoveries and relative percent differences between MS recoveries should be within the specified limits. DoD Perchlorate Handbook limits are 80-120%; RPD≤15%. The laboratory limits are 80-120%; RPD≤15%.

- No site MS/MSD was spiked with this SDG; therefore, was not evaluated. See Laboratory Control Sample (Section VII) for lab accuracy statement.

IX-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for perchlorate analysis in this SDG; therefore, was not evaluated.

X-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever was greater) was qualified as estimated, "J."

Sample: 54MW08 (L12080249-07), Perchlorate

$Y = mX + b$

Y = Response Ratio = Sample Area/Area Internal Standard O18LP
m = slope of curve
X = Amount Ratio = Conc. Analyte/Conc. Internal Std.
b = Y-intercept

Given:
m = 1.26
b = 0.000333
Y = Area = 35700/361000 = 0.0989
X = 0.07822

Conc. µg/L = (Ax * Cis * DF)
where: Conc. = Sample concentration in µg/L
Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard
Cis = Conc. Of internal Standard (µg/L)
DF = Dilution factor

Conc. µg/L = (0.07822 * 5 * 1) = 0.391 µg/L (Signal #1)
Reported Value = 0.392 µg/L
% Difference = 0.3%
Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

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Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-01	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW09	**Prep Method:**	6850	**Prep Date:**	08/16/2012 19:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406597	**Analyst:**	JWR	**Run Date:**	08/17/2012 04:23
Collect Date:	08/07/2012 10:25	**Dilution:**	1	**File ID:**	1LM.LM17340
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-03	**PrePrep Method:**	N/A	**Instrument:**	LCMS1
Client ID:	54MW11	**Prep Method:**	6850	**Prep Date:**	08/16/2012 19:30
Matrix:	Water	**Analytical Method:**	6850	**Cal Date:**	07/23/2012 19:47
Workgroup #:	WG406597	**Analyst:**	JWR	**Run Date:**	08/17/2012 04:42
Collect Date:	08/07/2012 11:40	**Dilution:**	1	**File ID:**	1LM.LM17341
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0		U	0.200	0.100

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-07
Client ID: 54MW08
Matrix: Water
Workgroup #: WG406597
Collect Date: 08/07/2012 12:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 19:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 05:01
File ID: 1LM.LM17342

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.392		0.200	0.100

Sample #: L12080249-09
Client ID: 54MW10
Matrix: Water
Workgroup #: WG406597
Collect Date: 08/07/2012 13:40
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 19:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 05:20
File ID: 1LM.LM17343

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	3.74		0.200	0.100

Sample #: L12080249-11
Client ID: 54MW13
Matrix: Water
Workgroup #: WG406597
Collect Date: 08/07/2012 15:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 6850
Analytical Method: 6850
Analyst: JWR
Dilution: 1
Units: ug/L

Instrument: LCMS1
Prep Date: 08/16/2012 19:30
Cal Date: 07/23/2012 19:47
Run Date: 08/17/2012 05:39
File ID: 1LM.LM17344

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.627		0.200	0.100

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L12080249

DATE: May 2, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 7, 2012. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of eight aqueous samples (includes three confirmation re-analysis samples) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L12080249-01	54MW10	L12080249-09
54MW11	L12080249-03	54MW10	L12080249-09RE
54MW08	L12080249-07	54MW13	L12080249-11
54MW08	L12080249-07RE	54MW13	L12080249-11RE

Data were reviewed by Eric Malarek and validated using a combination of project QAPP, *Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010) (DoD QSM), method-specific criteria, and laboratory SOP criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Blank Analysis
	X	Initial Calibration
	X	Continuing Calibration
	X	System Monitoring Compounds
	X	Laboratory Control Sample
	X	Matrix Spike/Spike Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

5/2/13

Date

RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L12080249

I-Holding Times and Preservation

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. Holding time criteria: For aqueous samples, explosive compounds are shipped cooled (@4°C ± 2°C) with a maximum holding time of 7 days from sample collection to preparative extraction and 40 days from preparative extraction to determinative analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/07/12, the cooler was received on 08/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: The aqueous samples were collected on 08/07/12. The samples were extracted on 08/10/12 and were analyzed on 08/14/12 and 08/16/12 for explosives. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Blank Analysis

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory or field activities. One method blank per analytical batch must be run on each instrument used to analyze samples. No contaminants should be present in the blanks. The DoD QSM criterion specifies all concentrations should be less than one-half MRL. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤5 times (5x) the maximum amount for explosive target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. µg/L	Action Level µg/L	B qualified samples (For this SDG)
08/13/12	WG406017-02	All target explosives <½MRL	NA	NA	None
08/15/12	WG406017-02	All target explosives <½MRL	NA	NA	None
08/15/12	54RB080812	All target explosives <½MRL	NA	NA	None

NA = Not Applicable
MRL = Method Reporting Limit
MDL = Method Detection Limit

III-Initial Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. The initial 5-point calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. The DoD QSM specifies that the correlation coefficient must be ≥0.995, the coefficient of determination ≥0.99, and/or the percent relative standard deviation (%RSD) must be ≤20%.

- For the explosives initial calibration performed on 02/23/12 on instrument HPLC5, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Samples 54MW09 (L12080249-01), 54MW11 (L12080249-03), 54MW08 (L12080249-07), 54MW10 (L12080249-09), and 54MW13 (L12080249-11) apply to this initial calibration.

- For the explosives initial calibration performed on 02/15/12 on instrument HPLC4, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with correlation coefficients ≥0.995. No qualifiers were applied. Confirmation samples 54MW08 (L12080249-07RE), 54MW10 (L12080249-09RE), and 54MW13 (L12080249-11RE) apply to this initial calibration.

IV-Continuing Calibration
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for the target compounds. Continuing calibration standards are analyzed after every 10 injections, and at the end of the analysis sequence. The DoD QSM specifies that the percent difference (%D) from initial calibration should be no greater than ±20%.

- For explosives initial calibration verification performed on 02/23/12 @16:22 on instrument HPLC5, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 08/13/12 @19:43 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/14/12 @04:29 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW09 (L12080249-01) and 54MW11 (L12080249-03) were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/14/12 @13:14 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW08 (L12080249-07), 54MW10 (L12080249-09), and 54MW13 (L12080249-11) were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/14/12 @20:03 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives initial calibration verification performed on 02/15/12 @20:05 on instrument HPLC4, all criteria were met for target compounds. No qualifiers were applied. No confirmation samples were reported using this initial verification calibration.

- For explosives continuing calibration performed on 08/15/12 @09:30 on instrument HPLC4, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/15/12 @21:18 on instrument HPLC4, all criteria were met. No qualifiers were applied. Confirmation samples 54MW08 (L12080249-07RE), 54MW10 (L12080249-09RE), and 54MW13 (L12080249-11RE) were reported using this continuing calibration.

- For explosives continuing calibration performed on 08/16/12 @07:12 on instrument HPLC4, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

V-System Monitoring Compound (Surrogates)
Laboratory performance on individual samples is established by means of spiking activities. The percent recovery (%R) for all samples and blanks must be within the laboratory control limits. DoD surrogate recovery limits are specified in Table G-3 of the DoD QSM (DoD, 2010). If the surrogate is not listed, then the laboratory criteria shall be used.

Aqueous Criteria: 1,2-dinitrobenzene (50-150%)

- All criteria were met for all runs. No qualifiers were applied.

VI-Laboratory Control Sample
Laboratory control samples (LCS) are used to monitor long-term accuracy of the analytical method. The analysis is performed at a frequency of 1 per analytical batch. The percent recoveries (%Rs) must be within the laboratory control limits. DoD QSM aqueous LCS recovery limits are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used.

- Sample WG406017-03 was used as the aqueous LCS for the explosives analysis on 08/13/12. All criteria were met. Samples 54MW09 (L12080249-01), 54MW11 (L12080249-03), 54MW08 (L12080249-07), 54MW10 (L12080249-09), and 54MW13 (L12080249-11) apply to this LCS.

- Sample WG406017-03 was used as the aqueous LCS for the explosives analysis on 08/15/12. All criteria were met. Samples 54MW08 (L12080249-07RE), 54MW10 (L12080249-09RE), and 54MW13 (L12080249-11RE) apply to this LCS.

VII-Matrix Spike/Matrix Spike Duplicate
Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The percent recoveries (%Rs) and the relative percent difference (RPD) must be within the laboratory control limits. DoD QSM aqueous MS/MSD recovery limits follow the LCS criteria and are specified in Table G-12 of the DoD QSM (DoD, 2010). If the compound is not listed, then the laboratory criteria shall be used. The DoD QSM aqueous precision limit for explosives is ≤20% RPD.

- No site specific aqueous MS/MSD was analyzed for explosives analysis in this SDG; therefore, was not evaluated. See LCS Section VI for laboratory accuracy statements.

VIII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 50% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for explosives analysis in this SDG; therefore, was not evaluated.

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters. The calculation was based calibration factors. The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. All positive values must have less than or equal to 40% RPD between the primary and secondary columns.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).

- For samples 54MW10 (L12080249-09) and 54MW10 (L12080249-09RE), 4-amino-2,6-dinitrotoluene (46.8%) was outside confirmation criteria and was qualified estimated "J" based upon this outlier.

- For sample 54MW13 (L12080249-11) and 54MW13 (L12080249-11RE), 4-amino-2,6-dinitrotoluene (111%) and RDX (98%) were outside confirmation criteria and were qualified estimated "J" based upon these outliers.

- For all other samples in this SDG, all positive detections met confirmation criteria.

Sample: 54MW08 (L12080249-07), 2-amino-4,6-dinitrotoluene

$Y = mX + b$

Y = Area of target compound for sample
m = slope of curve
X = Amount on column
b = Y-intercept

Given:
m = 0.38505705
b = -0.5967775
Y = Area = 14.38962

X = 38.92

Conc. µg/L = (Ax * Vt * DF) / (Vs)
where: Conc. = Sample concentration in µg/L
Ax = Amount of compound being measured (µg/L).
Vt = Volume of total extract (mL) from bench sheet.
Vs = Volume of sample extracted (mL).
DF = Dilution factor

Conc. µg/L = (38.92 * 10 * 1) / (970) = 0.401 µg/L (Signal #1)

Reported Value = 0.401 µg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Organic Data Review* (September 1994).

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Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-01
Client ID: 54MW09
Matrix: Water
Workgroup #: WG406134
Collect Date: 08/07/2012 10:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 08/10/2012 08:15
Cal Date: 02/23/2012 15:43
Run Date: 08/14/2012 10:20
File ID: 5L008172.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.03	0.258
2,4-Dinitrotoluene	121-14-2		U	1.03	0.258
2,6-Dinitrotoluene	606-20-2		U	1.03	0.258
2,4,6-Trinitrotoluene	118-96-7		U	1.03	0.258
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.03	0.258
2-Nitrotoluene	88-72-2		U	1.03	0.258
4-Nitrotoluene	99-99-0		U	1.03	0.258
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.03	0.258
RDX	121-82-4		U	1.03	0.258
Nitroglycerin	55-63-0		U	1.03	0.258

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	95.2	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-03	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW11	**Prep Method:**	3535	**Prep Date:**	08/10/2012 08:15
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406134	**Analyst:**	ECL	**Run Date:**	08/14/2012 10:59
Collect Date:	08/07/2012 11:40	**Dilution:**	1	**File ID:**	5L008173.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7		U	1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.04	0.260
RDX	121-82-4		U	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	104	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-07
Client ID: 54MW08
Matrix: Water
Workgroup #: WG406134
Collect Date: 08/07/2012 12:45
Sample Tag: CF01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC4
Prep Date: 08/10/2012 08:15
Cal Date: 02/15/2012 19:12
Run Date: 08/16/2012 02:36
File ID: 4L024250.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.03	0.258
2,4-Dinitrotoluene	121-14-2		U	1.03	0.258
2,6-Dinitrotoluene	606-20-2		U	1.03	0.258
2,4,6-Trinitrotoluene	118-96-7		U	1.03	0.258
2-Amino-4,6-dinitrotoluene	35572-78-2	0.516	J	1.03	0.258
2-Nitrotoluene	88-72-2		U	1.03	0.258
4-Nitrotoluene	99-99-0		U	1.03	0.258
4-Amino-2,6-dinitrotoluene	19406-51-0	0.415	J	1.03	0.258
RDX	121-82-4		U	1.03	0.258
Nitroglycerin	55-63-0		U	1.03	0.258

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	102	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

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Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-07
Client ID: 54MW08
Matrix: Water
Workgroup #: WG406134
Collect Date: 08/07/2012 12:45
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 08/10/2012 08:15
Cal Date: 02/23/2012 15:43
Run Date: 08/14/2012 13:53
File ID: 5L008178.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.03	0.258
2,4-Dinitrotoluene	121-14-2		U	1.03	0.258
2,6-Dinitrotoluene	606-20-2		U	1.03	0.258
2,4,6-Trinitrotoluene	118-96-7		U	1.03	0.258
2-Amino-4,6-dinitrotoluene	35572-78-2	0.401	J	1.03	0.258
2-Nitrotoluene	88-72-2		U	1.03	0.258
4-Nitrotoluene	99-99-0		U	1.03	0.258
4-Amino-2,6-dinitrotoluene	19406-51-0	0.505	J	1.03	0.258
RDX	121-82-4		U	1.03	0.258
Nitroglycerin	55-63-0		U	1.03	0.258

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	101	50	150	

J	Estimated value ; the analyte concentration was less than the LOQ.
U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC5
Client ID:	54MW10	**Prep Method:**	3535	**Prep Date:**	08/10/2012 08:15
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/23/2012 15:43
Workgroup #:	WG406134	**Analyst:**	ECL	**Run Date:**	08/14/2012 14:32
Collect Date:	08/07/2012 13:40	**Dilution:**	1	**File ID:**	5L008179.F
Sample Tag:	01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.03	0.258
2,4-Dinitrotoluene	121-14-2		U	1.03	0.258
2,6-Dinitrotoluene	606-20-2		U	1.03	0.258
2,4,6-Trinitrotoluene	118-96-7	9.17		1.03	0.258
2-Amino-4,6-dinitrotoluene	35572-78-2	1.66		1.03	0.258
2-Nitrotoluene	88-72-2		U	1.03	0.258
4-Nitrotoluene	99-99-0		U	1.03	0.258
4-Amino-2,6-dinitrotoluene	19406-51-0	2.69	J	1.03	0.258
RDX	121-82-4	7.84		1.03	0.258
Nitroglycerin	55-63-0		U	1.03	0.258

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	105	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-09	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW10	**Prep Method:**	3535	**Prep Date:**	08/10/2012 08:15
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG406134	**Analyst:**	ECL	**Run Date:**	08/16/2012 03:30
Collect Date:	08/07/2012 13:40	**Dilution:**	1	**File ID:**	4L024251.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.03	0.258
2,4-Dinitrotoluene	121-14-2		U	1.03	0.258
2,6-Dinitrotoluene	606-20-2		U	1.03	0.258
2,4,6-Trinitrotoluene	118-96-7	9.33		1.03	0.258
2-Amino-4,6-dinitrotoluene	35572-78-2	1.67		1.03	0.258
2-Nitrotoluene	88-72-2		U	1.03	0.258
4-Nitrotoluene	99-99-0		U	1.03	0.258
4-Amino-2,6-dinitrotoluene	19406-51-0	1.67	J	1.03	0.258
RDX	121-82-4	6.40		1.03	0.258
Nitroglycerin	55-63-0		U	1.03	0.258

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	106	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L12080249-11
Client ID: 54MW13
Matrix: Water
Workgroup #: WG406134
Collect Date: 08/07/2012 15:25
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 3535
Analytical Method: 8330B
Analyst: ECL
Dilution: 1
Units: ug/L

Instrument: HPLC5
Prep Date: 08/10/2012 08:15
Cal Date: 02/23/2012 15:43
Run Date: 08/14/2012 15:11
File ID: 5L008180.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7	9.40		1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2	1.84		1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0	3.77	J	1.04	0.260
RDX	121-82-4	4.77	J	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	90.3	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-11	**PrePrep Method:**	N/A	**Instrument:**	HPLC4
Client ID:	54MW13	**Prep Method:**	3535	**Prep Date:**	08/10/2012 08:15
Matrix:	Water	**Analytical Method:**	8330B	**Cal Date:**	02/15/2012 19:12
Workgroup #:	WG406134	**Analyst:**	ECL	**Run Date:**	08/16/2012 04:23
Collect Date:	08/07/2012 15:25	**Dilution:**	1	**File ID:**	4L024252.F
Sample Tag:	CF01	**Units:**	ug/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.04	0.260
2,4-Dinitrotoluene	121-14-2		U	1.04	0.260
2,6-Dinitrotoluene	606-20-2		U	1.04	0.260
2,4,6-Trinitrotoluene	118-96-7	9.24		1.04	0.260
2-Amino-4,6-dinitrotoluene	35572-78-2	1.79		1.04	0.260
2-Nitrotoluene	88-72-2		U	1.04	0.260
4-Nitrotoluene	99-99-0		U	1.04	0.260
4-Amino-2,6-dinitrotoluene	19406-51-0	1.08	J	1.04	0.260
RDX	121-82-4	1.63	J	1.04	0.260
Nitroglycerin	55-63-0		U	1.04	0.260

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	95.4	50	150	

U	Analyte was not detected. The concentration is below the reported LOD.

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Eric.malarek@CBI.com

MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L12080249

DATE: May 2, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on August 7, 2012. Samples were analyzed for the anions chloride, nitrate, and sulfate using USEPA SW-846 9056. A total of five aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L12080249-01	54MW10	L12080249-09
54MW11	L12080249-03	54MW13	L12080249-11
54MW08	L12080249-07		

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	X	Laboratory Duplicate Sample
	X	Matrix Spike and Spike Duplicate
	X	Field Duplicate Sample
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

.

Eric Malarek, Chemist

5/2/13

Date

RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L12080249

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C and 28 days for sulfate and chloride and Cool to 4°C±2°C with H_2SO_4 to pH<2 and 2 days for nitrate. The dates and times were compared between the sample collection and laboratory analysis (USEPA criteria).

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 08/07/12, the cooler was received on 08/08/12 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C. All criteria were met. No qualifiers were applied.

- Holding Time Review: Samples were collected on 08/07/12. The samples were prepped and analyzed on 08/08/12 for chloride, sulfate, and nitrate analysis. Sample collection dates may be found on the attached form 1s. All criteria were met. No qualifiers were applied.

II-Initial and Continuing Calibration

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Anions: 1 – blank
5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$)
ICV/CCV (90-110%)
Method Reporting Limit (MRL) (75-125%)

- Chloride, sulfate, and nitrate analysis was calibrated on 07/26/12 using linear equation techniques. All coefficients of determinations were ≥0.99 for chloride, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW09 (L12080249-01), 54MW11 (L12080249-03), 54MW08 (L12080249-07), 54MW10 (L12080249-09), and 54MW13 (L12080249-11) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <2MDL for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, 2-butanone, and phthalate esters, or 5 times (5X) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank 54RB080812 (L12080279-05) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (for this SDG)
08/08/12	Chloride	ICB/CCBs	<LOD	NA	None
08/08/12	Sulfate	ICB/CCBs	<LOD	NA	None
08/08/12	Nitrate	ICB/CCBs	<LOD	NA	None
08/08/12	Chloride	WG405863-01	<½MRL	NA	None
08/08/12	Sulfate	WG405863-01	<½MRL	NA	None
08/08/12	Nitrate	WG405863-01	<½MRL	NA	None
08/09/12	Chloride	54RB080812	<½MRL	NA	None
08/09/12	Sulfate	54RB080812	<½MRL	NA	None
08/09/12	Nitrate	54RB080812	<½MRL	NA	None

LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample

The laboratory control sample (LCS) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS are generated to determine long-term accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Per DoD QSM, all LCS results must fall within the specified control limits:

Anions: 90-110% (DOD QSM = 80-120%)

- Sample WG405863-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 08/08/12. All criteria were met. No qualifiers were applied. Samples 54MW09 (L12080249-01), 54MW11 (L12080249-03), 54MW08 (L12080249-07), 54MW10 (L12080249-09), and 54MW13 (L12080249-11) apply to this LCS.

V-Duplicate Sample Analysis

Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. Per DoD QSM, RPDs must be within established control limits (≤20%RPD).

- No site lab duplicate was performed with this SDG; therefore, was not evaluated.

VI-Matrix Spike and Matrix Spike Duplicate
Matrix spikes (MSs) and MSDs are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples or preparatory batch of similar matrix. Per DoD QSM, MS/MSD recoveries and RPDs should be within the specified limits:

Anions: 90-110%; RPD≤20% (DOD QSM = 80-120%; RPD≤20%)

- No site MS/MSD was spiked with this SDG; therefore, was not evaluated. See Laboratory Control Sample (Section IV) for lab accuracy statement.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No site field groundwater sample duplicate pair was analyzed for anions analysis in this SDG; therefore, was not evaluated.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW09 (L12080249-01), sulfate

$Y = mX + b$

Y = Sample Area
m = slope of curve
X = Concentration (mg/L)
b = Y-intercept
DF = Dilution Factor

Given:
m = 0.1121
b = -0.0220
Y = Area = 3.234
DF = 3

X = 29.05 mg/L * DF = 31.40 mg/L * 3 = 87.1 mg/L

Reported concentration = 87.2 mg/L
%D = 0.1%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Form I Copy

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Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-01	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW09	**Prep Method:**	9056	**Prep Date:**	08/08/2012 18:28
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG405863	**Analyst:**	JBK	**Run Date:**	08/08/2012 22:09
Collect Date:	08/07/2012 10:25	**Dilution:**	3	**File ID:**	I20808122209.29
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	6.24		0.600	0.300
Nitrate	14797-55-8		U	1.80	0.300
Sulfate	14808-79-8	87.2		3.00	1.50

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-03	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW11	**Prep Method:**	9056	**Prep Date:**	08/08/2012 18:28
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG405863	**Analyst:**	JBK	**Run Date:**	08/08/2012 22:27
Collect Date:	08/07/2012 11:40	**Dilution:**	3	**File ID:**	I20808122227.30
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.40		0.600	0.300
Nitrate	14797-55-8		U	1.80	0.300
Sulfate	14808-79-8	97.6		3.00	1.50

U	Analyte was not detected. The concentration is below the reported LOD.

Form I Copy

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-07	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW08	**Prep Method:**	9056	**Prep Date:**	08/08/2012 18:28
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG405863	**Analyst:**	JBK	**Run Date:**	08/08/2012 22:46
Collect Date:	08/07/2012 12:45	**Dilution:**	1	**File ID:**	I20808122246.31
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.40		0.200	0.100
Nitrate	14797-55-8	0.581	J J	0.600	0.100
Sulfate	14808-79-8	41.8		1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-09	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW10	**Prep Method:**	9056	**Prep Date:**	08/08/2012 18:28
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG405863	**Analyst:**	JBK	**Run Date:**	08/08/2012 23:04
Collect Date:	08/07/2012 13:40	**Dilution:**	3	**File ID:**	I20808122304.32
Sample Tag:	DL01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	3.71		0.600	0.300
Nitrate	14797-55-8	0.594	J J	1.80	0.300
Sulfate	14808-79-8	102		3.00	1.50

J	Estimated value ; the analyte concentration was less than the LOQ.

Form I Copy

Microbac

Lab Report #: L12080249
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L12080249-11	**PrePrep Method:**	N/A	**Instrument:**	IC2
Client ID:	54MW13	**Prep Method:**	9056	**Prep Date:**	08/08/2012 18:28
Matrix:	Water	**Analytical Method:**	9056	**Cal Date:**	07/26/2012 14:25
Workgroup #:	WG405863	**Analyst:**	JBK	**Run Date:**	08/08/2012 23:22
Collect Date:	08/07/2012 15:25	**Dilution:**	1	**File ID:**	I20808122322.33
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.07		0.200	0.100
Nitrate	14797-55-8	0.681		0.600	0.100
Sulfate	14808-79-8	38.2		1.00	0.500

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MEMORANDUM

TO: Tim Leahy, Shaw E&I Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, Shaw E&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon Microbac Laboratories, Inc. L13050918

DATE: July 24, 2013

The purpose of this memorandum is to present the data validation report for the samples collected at Main Manufacturing Area at RFAAP for SWMU54 RFI/IM on May 16, 2013. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 415.1. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. A total of six aqueous samples were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW02	L13050918-01	54MW12	L13050918-04
54MW02	L13050918-02	54MW13	L13050918-05
54MW12	L13050918-03	54MW13	L13050918-06

Data were reviewed and validated using a combination of project QAPP, *DoD Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 25, 2010* (DoD, 2010), and method-specific criteria. The data qualifier scheme was consistent with the *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993). Parameters evaluated are presented in **Table 1**. Data associated with parameters in compliance with quality control specifications have not been qualified. Data associated with parameters that did not comply with quality control specifications and directly impacted project data have been qualified in accordance with USEPA Region III specifications.

Table 1 Laboratory Performance Criteria

Qualified		Parameter
Yes	No	
X		Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	Laboratory Control Sample and Laboratory Control Sample Duplicate
	X	Matrix Spike and Spike Duplicate
	X	Laboratory Duplicate
	X	Field Duplicate
X		Quantitation Verification and Data Review

The quality of data collected in support of this sampling activity is considered acceptable with noted qualifications.

Eric Malarek, Chemist

7/24/13
Date

RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13050918

I-Holding Times and Preservation

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample analysis. Holding time criteria: Cool 4°C±2°C, HCl pH<2, 28 days for TOC, TIC, DOC, and DIC (USEPA criteria). The dates and times were compared between the sample collection and laboratory analysis.

- Temperature Review: The temperature blank was sent with each cooler and recorded by the lab upon receipt. For samples collected on 05/16/13, the cooler was received on 05/17/13 by the primary laboratory (Microbac Laboratories, Inc.) at 11.0°C. All samples were detected for TOC, TIC, DOC, and DIC; therefore, were qualified estimated "L" for detections based upon this outlier.

- Holding Time Review: The samples were collected on 05/16/13. The TOC and TIC analysis were run on 05/23/13 and 05/24/13. The DOC and DIC analysis were run on 05/24/13. Sample collection dates may be found on the attached form 1s. All criteria were met.

II-Initial and Continuing Calibration

Bench and run summary sheets were reviewed to determine whether calibration was performed at the beginning of sample analysis using the following criteria. Percent recoveries for initial and continuing calibration (90-110%) must be within limits.

TOC, TIC, DOC, and DIC: 1 - blank
5 - standards ($r \geq 0.995$)
ICV/CCV (90-110%)

- The TOC and TIC analysis were run on 05/23/13 and 05/24/13. The DOC and DIC analysis were run on 05/24/13. The initial calibration for TC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The initial calibration for TIC was analyzed on 08/01/12 with a coefficient of determination of 0.9999. The ICV and CCVs were evaluated for where they bracketed reported samples. All ICV/CCVs that bracketed reported samples were within criteria. All criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-01), 54MW02 (L13050918-02), 54MW12 (L13050918-03), 54MW12 (L13050918-04), 54MW13 (L13050918-05), and 54MW13 (L13050918-06) apply to these initial and continuing calibrations.

III-Blank Analysis

The purpose of blank analyses is to determine the presence and magnitude of contamination problems resulting from field and laboratory activities. One method blank per analytical batch must be run on each instrument used to analyze samples. Calibration blanks were analyzed initially and at a 10% frequency thereafter for each batch. No contaminants should be detected in any of the associated blanks >MDL. The DoD QSM criteria specifies all concentrations should be less than ½ MRL (<MRL for common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters) and <LOD (i.e. <2MDL) for the calibration blanks. Positive sample results are reported and qualified "B", if the concentration of the compound in the sample is ≤10 times (10x) the maximum amount in any blank for the common laboratory contaminants methylene chloride, acetone, toluene, 2-butanone, and phthalate esters, or 5 times (5x) the maximum amount for other target compounds. **Table 2** summarizes the blank contamination analysis. Action levels are based upon dilution factor of one. The rinse blank RB051513 (L13050819-14) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank RB051513 (L13050819-13) applies to all total samples (TOC and TIC) in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	Analysis	QC Blank ID	Max Conc. mg/L	Action Level mg/L	B qualified samples (For this SDG)
05/23/13	TOC	ICB/CCBs	<LOD	NA	None
05/28/13	TOC	ICB/CCBs	<LOD	NA	None
05/23/13	TOC	WG431487-01	<½MRL	NA	None
05/23/13	DOC	WG431489-01	<½MRL	NA	None
05/24/13	TOC	WG431490-01	<½MRL	NA	None
05/23/13	TOC	RB051513	0.514J	2.57	54MW02, 54MW12, 54MW13
05/23/13	TIC	RB051513	<½MRL	NA	None
05/24/13	DOC	RB051513	3.09	15.5	54MW02, 54MW12, 54MW13
05/24/13	DIC	RB051513	<½MRL	NA	None

J = Estimated value.
LOD = Limit of Detection
MRL = Method Reporting Limit
MDL = Method Detection Limit
NA = Not Applicable

IV-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS and LCSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. All aqueous LCS results must fall within the control limits (85-115%; RPD≤15%).

- Samples WG431487-02 and WG431487-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 05/23/13. All criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-01) and 54MW12 (L13050918-03) apply to this LCS/LCSD.

- Samples WG431490-02 and WG431490-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 05/24/13. All criteria were met. No qualifiers were applied. Sample 54MW13 (L13050918-05) applies to this LCS/LCSD.

- Samples WG431489-02 and WG431489-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 05/23/13. All criteria were met. No qualifiers were applied. Samples 54MW02 (L13050918-02), 54MW12 (L13050918-04), and 54MW13 (L13050918-06) apply to this LCS/LCSD.

V-Matrix Spike and Spike Duplicate
Matrix spikes (MSs) and matrix spike duplicates (MSDs) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. Specific criteria include the analyses of matrix spike samples at a frequency of one MS/MSD per 20 samples of similar matrix. The percent recoveries (%Rs or RPD) must be within the specified control limits (85-115%; RPD≤15%).

- No site specific aqueous MS/MSD was performed for TOC and TIC analysis on 05/23/13 or 05/28/13; therefore, was not evaluated. See Section IV for lab accuracy statement using LCS/LCSD.

- No site specific aqueous MS/MSD was performed for DOC and DIC analysis on 05/23/13 or 05/24/13; therefore, was not evaluated. See Section IV for lab accuracy statement using LCS/LCSD.

VI-Laboratory Duplicate Sample Analysis
Duplicate sample determinations are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analysis is performed to generate data in order to determine the long-term precision of the analytical method on various matrices. RPDs must be within established control limits (≤30%RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statement using LCS/LCSD.

VII-Field Duplicate Sample Analysis
Field duplicates were collected to identify the cumulative precision of the sampling and analytical process and sent to the laboratory blind. The RPD was calculated only for those analytes which were detected at levels exceeding the method reporting limits in both samples of the duplicate pair. Analytes that were qualified rejected (R-qualified) in either sample of the duplicate pair were excluded from the duplicate assessment. Precision control criterion was established at 25% RPD for the aqueous samples.

- No aqueous field duplicate was analyzed for TOC, TIC, DOC, and DIC with this SDG; therefore, it was not evaluated.

VIII-Quantitation Verification and Data Review
The accuracy of analytical results is verified through the calculation of several parameters. The percent difference (%D) between the calculated and the reported values should be within 10%. The following calculations were performed for verification.

- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

- The total and dissolved fractions were compared. The organic and inorganic carbon data results for samples 54MW02 (L13050918-01), 54MW02 (L13050918-02), 54MW12 (L13050918-03), 54MW12 (L13050918-04), 54MW13 (L13050918-05), and 54MW13 (L13050918-06) are presented in **Table 3**. The samples with the odd fraction lab IDs are the total fractions and the samples with the even fraction lab IDs are the dissolved fractions. Data qualification was applied based upon professional judgment. Generally, if the concentrations are greater than the MRL and the dissolved concentration is greater than its total concentration by >10%D, then both results have been flagged as estimated with a "J." We are looking at the TOC/TIC to evaluate the immobilization potential of TNT and RDX. The DOC/DIC is a byproduct of organic compound oxidation and indicates the difference in microbial oxidation processes within and outside the plume area. If the samples exhibit continual microbial oxidation coupled with the time differences of sample preservation from sample collection (i.e. TOC in field and DOC at the lab), this might be the cause of total / dissolved imbalance here.

Table 3 Total/Dissolved Summary

Sample ID	Analyte	MRL (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW02	TOC/DOC	1.0; 2.0	1.68	1.64J	NA	None
54MW02	TIC/DIC	1.0; 2.0	17.1	57.5	70.3	J
54MW12	TOC/DOC	1.0; 2.0	1.36	2.37	42.6	J
54MW12	TIC/DIC	1.0; 2.0	31.6	67.4	53.1	J
54MW13	TOC/DOC	1.0; 2.0	0.817J	3.49	76.6	J
54MW13	TIC/DIC	1.0; 2.0	5.75	49.0	88.3	J

J = Estimated value.
MRL = Method Reporting Limit
NA = Not Applicable

Sample: 54MW02 (L13050918-01), TOC

TC: Y = m*X (mg/L) + b

m = 38.46
b = 2.031
Y = 724.6
DF = 1

X = (18.79 mg/L) * 1 = 18.79 mg/L

TIC: Y = m*X (mg/L) + b

m = 29.16
b = 6.703
Y = 505.6
DF = 1

X = (17.11 mg/L) * 1 = 17.11 mg/L

TOC (mg/L) = TC (mg/L) – TIC (mg/L) = 18.79 – 17.11 = 1.68 mg/L

Reported Value = 1.68 mg/L
% Difference = 0.0%
Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers[1]	
No Code	Confirmed identification.
U	Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimation.
B	Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank.
N	Non-target analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q	One or more quality control criteria failed.
USEPA Region III Data Validation Qualifiers[2]	
R	Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
B	Not detected substantially above the level of the reported in laboratory or field blanks.
J	Analyte present. Reported value may not be accurate or precise.
UJ	Not detected, quantitation limit may be inaccurate or imprecise.
N	Tentative Identification. Consider present. Special methods may be to confirm its presence or absence in future sampling efforts.
NJ	Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher.
UL	Not detected, quantitation limit is probably higher.

[1]The noted laboratory qualifiers are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. Data qualifiers may be combined when appropriate. Ref.: *DOD Quality Systems Manual for Environmental Laboratories, Final Version 4.2* (DoD, 2010).

[2]The USEPA data validation qualifiers are referenced from *Region III Modifications to the National Functional Guidelines for Inorganic Data Review* (April, 1993).

Microbac

Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13050918-01 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW02 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431487 | **Analyst:** BAF | **Run Date:** 05/23/2013 22:40
Collect Date: 05/16/2013 07:10 | **Dilution:** 1 | **File ID:** TC05232013.028
Sample Tag: 01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		17.1	J	1.00	0.500

Sample #: L13050918-01 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW02 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431487 | **Analyst:** BAF | **Run Date:** 05/23/2013 22:40
Collect Date: 05/16/2013 07:10 | **Dilution:** 1 | **File ID:** TC05232013.028
Sample Tag: 01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.68	B	1.00	0.500

Sample #: L13050918-02 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW02 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431489 | **Analyst:** BAF | **Run Date:** 05/24/2013 02:54
Collect Date: 05/16/2013 07:10 | **Dilution:** 2 | **File ID:** TC05232013.048
Sample Tag: DL01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		57.5	J	2.00	1.00

Sample #: L13050918-02 | **PrePrep Method:** N/A | **Instrument:** TOC-VWP
Client ID: 54MW02 | **Prep Method:** 415.1 | **Prep Date:** N/A
Matrix: Water | **Analytical Method:** 415.1 | **Cal Date:** 08/06/2012 08:11
Workgroup #: WG431489 | **Analyst:** BAF | **Run Date:** 05/24/2013 02:54
Collect Date: 05/16/2013 07:10 | **Dilution:** 2 | **File ID:** TC05232013.048
Sample Tag: DL01 | **Units:** mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.64	J B	2.00	1.00

J	Estimated value ; the analyte concentration was less than the LOQ.

Certificate of Analysis

Sample #: L13050918-03 | PrePrep Method: N/A | Instrument: TOC-VWP
Client ID: 54MW12 | Prep Method: 415.1 | Prep Date: N/A
Matrix: Water | Analytical Method: 415.1 | Cal Date: 08/06/2012 08:11
Workgroup #: WG431487 | Analyst: BAF | Run Date: 05/23/2013 22:53
Collect Date: 05/16/2013 08:20 | Dilution: 1 | File ID: TC05232013.029
Sample Tag: 01 | Units: mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.36	B	1.00	0.500

Sample #: L13050918-03 | PrePrep Method: N/A | Instrument: TOC-VWP
Client ID: 54MW12 | Prep Method: 415.1 | Prep Date: N/A
Matrix: Water | Analytical Method: 415.1 | Cal Date: 08/06/2012 08:11
Workgroup #: WG431487 | Analyst: BAF | Run Date: 05/23/2013 22:53
Collect Date: 05/16/2013 08:20 | Dilution: 1 | File ID: TC05232013.029
Sample Tag: 01 | Units: mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		31.6	J	1.00	0.500

Sample #: L13050918-04 | PrePrep Method: N/A | Instrument: TOC-VWP
Client ID: 54MW12 | Prep Method: 415.1 | Prep Date: N/A
Matrix: Water | Analytical Method: 415.1 | Cal Date: 08/06/2012 08:11
Workgroup #: WG431489 | Analyst: BAF | Run Date: 05/24/2013 03:08
Collect Date: 05/16/2013 08:20 | Dilution: 2 | File ID: TC05232013.049
Sample Tag: DL01 | Units: mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.37	B	2.00	1.00

Sample #: L13050918-04 | PrePrep Method: N/A | Instrument: TOC-VWP
Client ID: 54MW12 | Prep Method: 415.1 | Prep Date: N/A
Matrix: Water | Analytical Method: 415.1 | Cal Date: 08/06/2012 08:11
Workgroup #: WG431489 | Analyst: BAF | Run Date: 05/24/2013 03:08
Collect Date: 05/16/2013 08:20 | Dilution: 2 | File ID: TC05232013.049
Sample Tag: DL01 | Units: mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		67.4	J	2.00	1.00

Microbac

Lab Report #: L13050918
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13050918-05	**PrePrep Method:**	N/A	**Instrument:**	TOC-VWP
Client ID:	54MW13	**Prep Method:**	415.1	**Prep Date:**	N/A
Matrix:	Water	**Analytical Method:**	415.1	**Cal Date:**	08/06/2012 08:11
Workgroup #:	WG431490	**Analyst:**	BAF	**Run Date:**	05/24/2013 06:30
Collect Date:	05/16/2013 09:35	**Dilution:**	1	**File ID:**	TC05232013.066
Sample Tag:	01	**Units:**	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.817	J B	1.00	0.500

J	Estimated value ; the analyte concentration was less than the LOQ.

Certificate of Analysis

Sample #: L13050918-05
Client ID: 54MW13
Matrix: Water
Workgroup #: WG431490
Collect Date: 05/16/2013 09:35
Sample Tag: 01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 1
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/24/2013 06:30
File ID: TC05232013.066

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		5.75	J	1.00	0.500

Sample #: L13050918-06
Client ID: 54MW13
Matrix: Water
Workgroup #: WG431489
Collect Date: 05/16/2013 09:35
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/24/2013 03:43
File ID: TC05232013.052

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		49.0	J	2.00	1.00

Sample #: L13050918-06
Client ID: 54MW13
Matrix: Water
Workgroup #: WG431489
Collect Date: 05/16/2013 09:35
Sample Tag: DL01

PrePrep Method: N/A
Prep Method: 415.1
Analytical Method: 415.1
Analyst: BAF
Dilution: 2
Units: mg/L

Instrument: TOC-VWP
Prep Date: N/A
Cal Date: 08/06/2012 08:11
Run Date: 05/24/2013 03:43
File ID: TC05232013.052

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.49	B	2.00	1.00