

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

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

HOLE DIAMETER: 2"

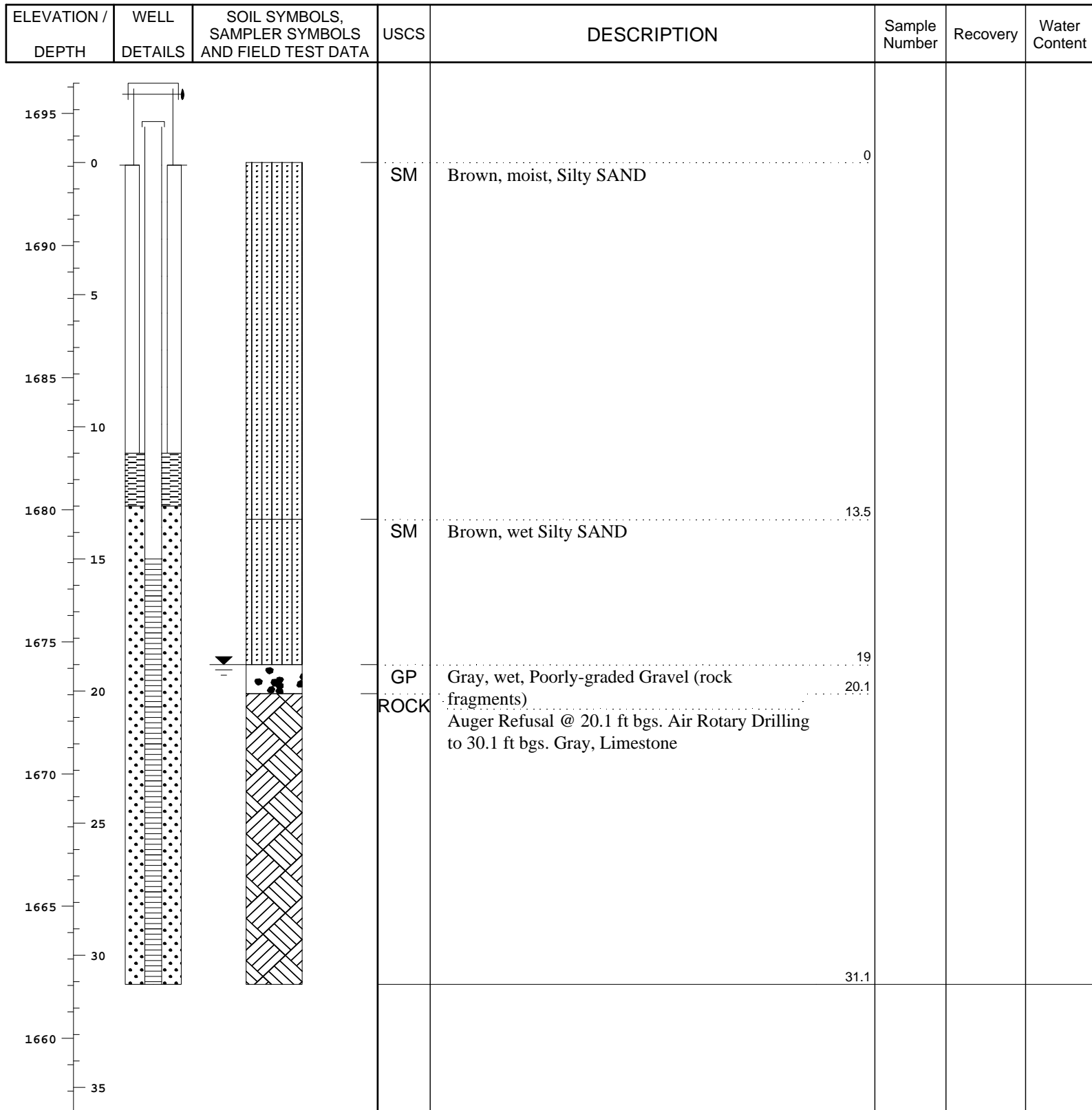


FIGURE NO.

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

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

HOLE DIAMETER: 2"

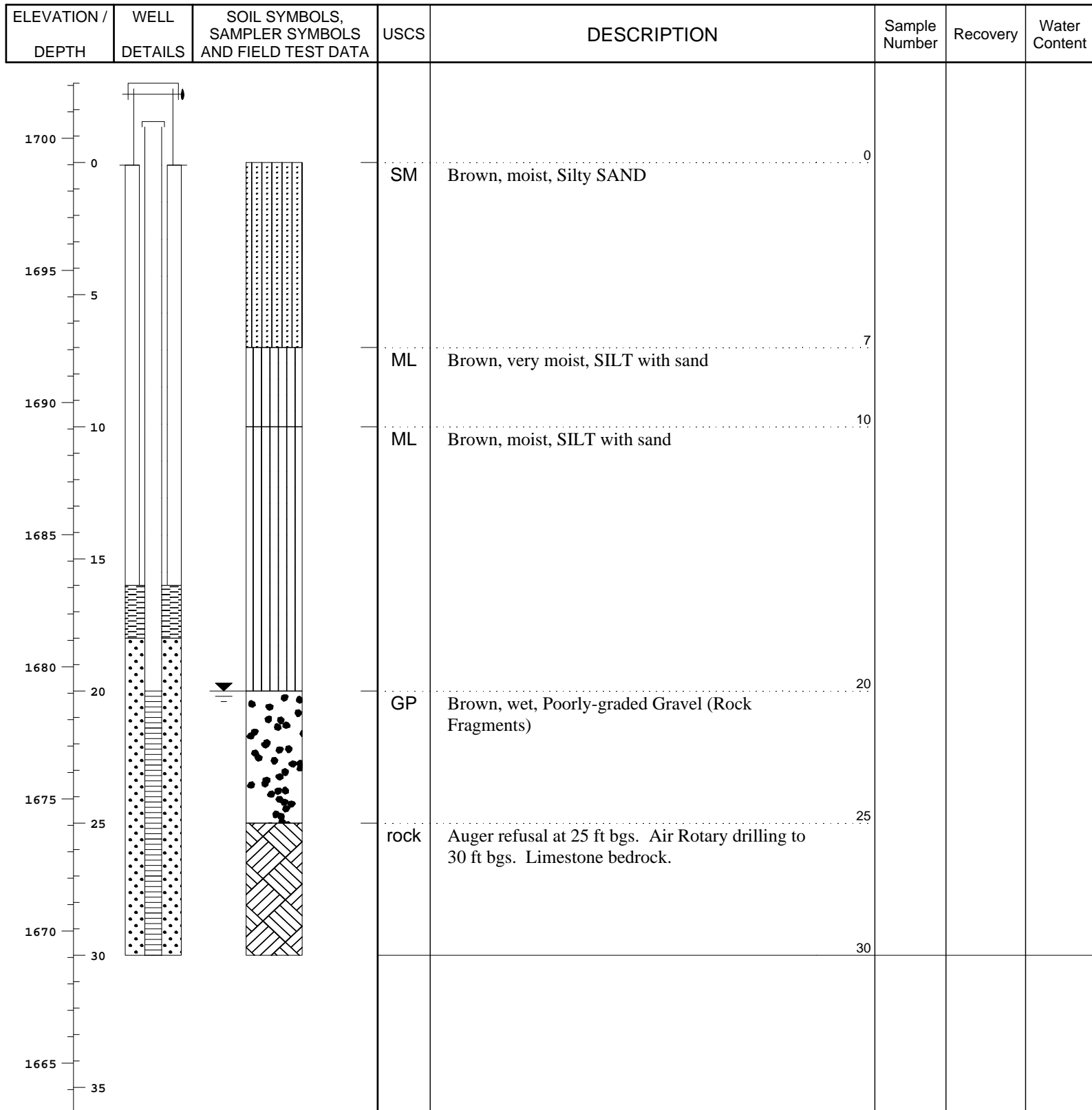


FIGURE NO.

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

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

HOLE DIAMETER: 2"

ELEVATION / DEPTH	WELL DETAILS	SOIL SYMBOLS, SAMPLER SYMBOLS AND FIELD TEST DATA	USCS	DESCRIPTION	Sample Number	Recovery	Water Content
1700 0							
			FILL	Black, dry, FILL (Road Gravel)	0		
			SM	Light brown, very moist, Silty SAND	1		
1695 5					5		
			SP	Light brown, moist, Poorly-graded SAND with silt seams; fining downward to Silty SAND			
1690 10							
1685 15							
			SC	Gray/brown, wet Clayey SAND	16.5		
			GP	Brown, wet, Silty SAND	17		
1680 20				Brown, wet, Poorly-graded Gravel (Rock Fragments)			
					22		
1675 25				Auger Refusal @ 22.0 ft bgs			
1670 30							
1665 35							

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

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

HOLE DIAMETER: 2"

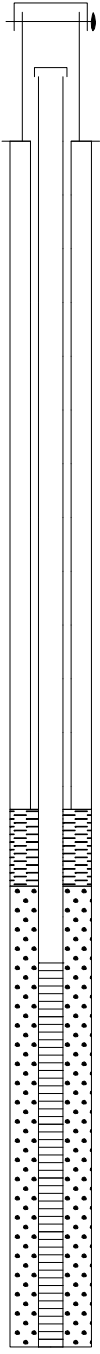
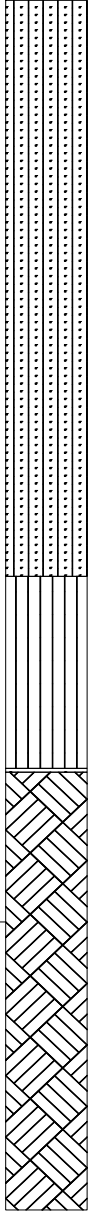
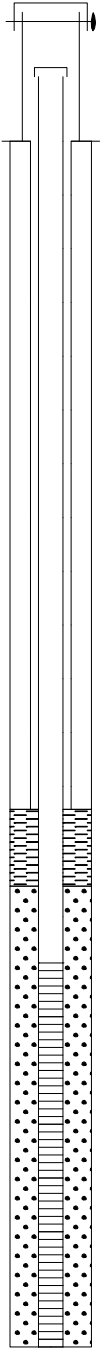
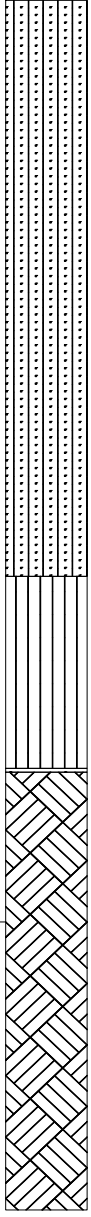
ELEVATION / DEPTH	WELL DETAILS	SOIL SYMBOLS, SAMPLER SYMBOLS AND FIELD TEST DATA	USCS	DESCRIPTION	Sample Number	Recovery	Water Content
1700			SM	Brown, moist, Silty SAND	0		
1695							
1690							
1685							
1680			ML	Brown, wet, Sandy SILT	15		
1675			ROCK	Auger Refusal @ 20.0 ft bgs. Air Rotary drilling to 31.5 ft bgs. Grey Limestone	20 20.1		
1670							
1665							
				Well Set @ 31.5 ft bgs	31.5		

FIGURE NO.

Appendix A-2
Field Sampling Forms

S. 16.13
S. Hillebrand
G. Tucker

Well sumw13 stabilized @ 9:35.

Samples collected.

FC = 4.2 MW = 1.5

Pump pulled 9 MOB off site.

IPW taken to treatment plant.

8.5.13
S. Hillebrand
S. Korbak

Arrived onsite @ 7:00 to get bogged in.

Bogges were received @ 7:30. MOB to sumw13
for safety meetings w/ BAE along with our Jett
tailgate meeting. Honda was standardized during meetings.
A barrel of GW levels was collected.

MW1	18.64	MW8 ⁺	12.00
MW2	19.96	MW9 ⁺	16.94
MW3	21.51	MW10 ⁺	14.31
MW4	15.62	MW11 ⁺	16.62
MW5	15.81	MW12	20.68
MW6	Dry	MW13	18.92
MW7	13.60	MW14	20.12

MOB to sumw13. Set up equipment, lowered
pump to 23'. Began pumping @ 8:30. Pump set
@ 200 mL/min w/ discharge going through flow through
cell and out to a 5 gal container

sumw13

Time	Temp	Cond	DO	pH	OP	turb	T
8:35	14.27	.327	6.85	6.56	121	1.5	18.99
8:40	14.24	.332	6.82	6.56	127	0.0	19.01
8:45	13.91	.344	6.72	6.60	136	0.0	19.03
8:50	13.89	.349	6.67	6.60	139	0.2	19.03
8:55	13.90	.349	6.55	6.59	141	0.2	19.04

8.5.13
JH
SK

Time	Temp	cond	DO	pH	orp	turb	Fe
9:00	13.90	.350	6.56	6.58	144	0.0	19.04
9:05	13.89	.350	6.57	6.58	145	0.2	19.05
9:30	13.89	.351	6.57	6.57	146	0.2	19.05

well stabilized @ 9:10. Sample collected @ 9:10

Fe sample and Mn sample collected,

Fe = <.2 Mn = .15 Decomed pump.

MOB to 54mw06 to recover water level.

Water level still up. No mud on tip of pole.

MOB to 54mw1. Set well @ 45'. Pumped

well @ 200m/h. Well started @ 9:55.

Time	Temp	cond	DO	pH	orp	turb	Fe
10:00	14.15	.398	5.95	7.38	115	0.2	16.71
10:03	14.47	.399	4.41	7.35	118	2.3	16.71
10:10	14.41	.398	4.26	7.38	118	2.1	16.71
10:15	14.34	.400	4.10	7.37	119	2.0	16.72
10:20	14.46	.397	4.17	7.38	120	2.1	16.72
10:25	14.41	.399	4.03	7.38	121	2.0	16.72
10:30	14.50	.397	4.11	7.38	121	1.0	16.72
10:35	14.46	.397	4.13	7.38	122	1.0	16.72
10:40	13.43	.398	4.13	7.38	122	1.2	16.72

Well stabilized @ 10:40. Samples collected @

10:40. Fe = <.2 Mn = .48

Decomed supply & MOB to 54mw04

54mw01 collected.

54mw04

8.5.13
JH
SK

Time	Temp	cond	DO	pH	orp	turb	Fe
11:30	13.67	.968	6.82	6.94	63	2.0	15.66
11:35	12.99	.971	0.59	6.93	24	1.2	15.69
11:40	13.66	.964	0.50	6.93	8	13.0	15.71
11:45	13.28	.959	0.44	6.94	3	10.1	15.73
11:50	13.14	.949	0.44	6.95	-6	0.0	15.73
11:55	13.24	.941	0.42	6.95	-9	0.0	15.73
12:00	13.20	.940	0.43	6.95	-12	0.0	15.73
12:05	13.21	.938	0.41	6.96	-17	0.0	15.74
12:10	13.22	.937	0.41	6.96	-18	0.0	15.74
12:15	13.22	.937	0.39	6.96	-18	0.0	15.74

Well stabilized @ 12:15. Samples collected

@ 12:15. Fe = <.2 Mn = .5^{high?} retest Mn = .05

Decom supplies. MOB to 54mw14.

well set @ 25'. Pump started @ 12:55. 200m/h

Time	Temp	cond	DO	pH	orp	turb	Fe
13:00	13.52	.582	.82	6.90	55	1.0	20.21
13:05	13.70	.582	.48	6.88	37	1.1	20.24
13:10	13.30	.581	.47	6.89	32	7.4	20.26
13:15	13.16	.578	.52	6.89	33	5.0	20.27
13:20	13.22	.556	.51	6.86	50	0.0	20.27
13:25	13.20	.554	.52	6.84	51	0.0	20.27
13:30	13.21	.554	.51	6.84	52	0.0	20.28
13:35	13.21	.554	.52	6.84	50	0.0	20.28

8.5.13
JH
SK

sample collected @ 1335. sumu4 taken
 $Fe = .2$ $Mn = .5$

Person supplies & mob to sumuor.

Each bit for Mn has only 1 more packet.

More were ordered for overnight delivery.

mob from site to stage drums for
tomorrow disposal.

8.6.13
JH
SK

Arrived onsite @ 6:00 to meet with
surveyor. Rodeged sumuor & mob to
site. Surveyed 49mw04, 49mw03, & 49mw03
Before 8:45. Equipment failure caused us to
demob & the surveyor to return to his
office for replacement equipment. Arrived back
onsite 10:30. Disposed of $\approx 200g$ of water
from the drums onsite from sumu4 sampling
and development. ≈ 15 gallons IOW Disposed from sumu4

8-7-13
ON
ST

met & share at the hotel @ 6:30. Held
tailgate safety meeting and JSA. Mob to site.
Standardized horiba @ the site while
waiting on the guards to get us behind
gate #1E. Call placed @ 7:30. Guard let us
in @ 8:45. Pump intake set @ 23'. Pump
turned on @ 9:00. 200 ml/m

54MW09

Time	Temp	Cond	DO	pH	orp	Turb	♀
9:05	14.38	.517	2.21	7.26	-154	121	16.96
9:10	13.70	.468	2.15	7.16	-137	111	16.98
9:15	13.25	.446	2.29	7.10	-99	97.0	16.98
9:20	13.31	.441	2.32	7.04	-78	38.3	16.98
9:25	13.26	.436	2.38	7.10	-53	13.8	16.99
9:30	13.25	.429	2.39	7.12	-36	6.8	16.99
9:35	13.26	.423	2.38	7.12	-22	5.3	16.99
9:40	13.25	.420	2.38	7.12	-16	5.1	17.01
9:45	13.25	.418	2.37	7.12	-14	5.3	17.01
9:50	13.25	.419	2.36	7.12	-13	6.1	17.01

Samples collected @ 9:50.

Fe = 2 mm = .05

Recon supplies & more to 54MW11.

8-7-13
ON
ST

Set up on 54MW11 @ 10:20.
Well set @ 25'. Pump turned on @ 10:25.
250 ml/m.

Time	Temp	Cond	DO	pH	orp	Turb	♀
10:25	13.43	.639	4.78	7.03	57	1.3	16.65
10:30	13.57	.632	5.01	7.03	61	0.0	16.66
10:35	13.54	.636	4.70	7.03	65	0.0	16.66
10:40	13.55	.638	4.68	7.03	66	0.0	16.66
10:45	13.48	.638	4.64	7.03	68	0.0	16.66
10:50	13.46	.637	4.66	7.04	68	0.0	16.67
10:55	13.46	.637	4.65	7.04	68	0.0	16.67

well stabilized @ 10:55. Samples collected at
10:55. Fe = 2 mm = .05.

Recon pump & more to 54MW08.

Set up on 54MW08 @ 11:20

Pump set @ 25'. Pump turned on @ 11:25

200 ml/m.

Time	Temp	Cond	pH	DO	orp	turb	♀
11:30	13.59	.566	6.97	1.46	67	0.0	12.03
11:35	13.44	.568	6.96	.71	58	0.0	12.04
11:40	13.42	.567	6.96	.68	46	0.0	12.04
11:45	13.43	.564	6.96	.19	38	0.0	12.05
11:50	13.44	.560	6.96	.67	37	0.0	12.05
11:55	13.43	.558	6.95	.68	41	0.0	12.06
12:00	13.40	.558	6.95	.68	42	0.0	12.06
12:05	13.40	.559	6.95	.67	42	0.0	12.06

87.03
5A
SF

Well stabilized @ 12:05. Samples collected @

12:05. Fe = 2.2 Mn = 2.05.

Decom applies 9 moB to 54mw10.

Well set @ 12:40. Pump turned on

@ 12:45. Set @ 22'. 300ml/min

Time	Temp	cond	DO	pH	orp	turb	Σ
1245	14.42	.494	.84	6.46	48	0.0	14.33
1250	14.44	.495	.79	6.47	46	0.0	14.34
1255	14.50	.495	.80	6.47	41	0.0	14.33
1300	14.49	.496	.89	6.47	39	0.0	14.33
1305	14.51	.495	.86	6.48	37	0.0	14.34
1310	14.51	.496	.87	6.48	36	0.0	14.33
1315	14.51	4.96	.86	6.48	35	0.0	14.33

Well stabilized @ 13:15. Samples collected.

Fe < 2.2 Mn < 0.5. Gate guards called

prior to sample collection to let us back in the gate. Decommed equipment.

Did more blanks while waiting for guard.

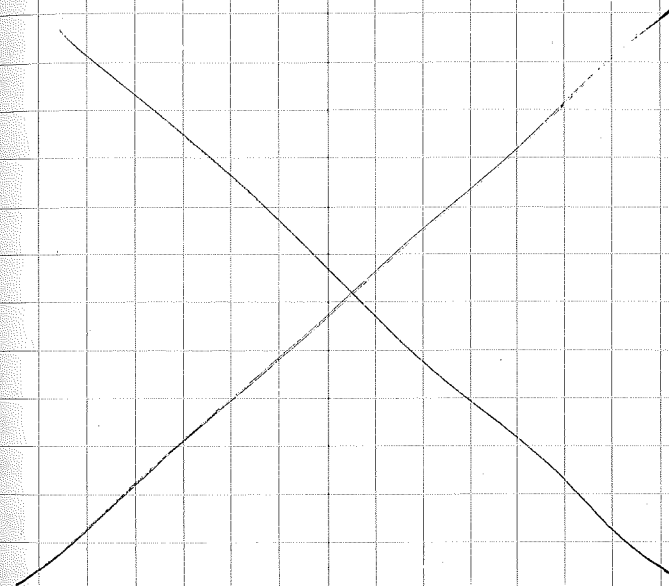
54RB080713 @ 1335

MOB to 54mw12 well set @ 26'. 300ml/min started @ 1345.

54mw12

Time	Temp	cond	DO	pH	orp	turb	Σ
1345	15.56	.547	2.89	6.4	89	54	20.71
1350	15.87	.563	2.81	6.37	88	13	20.74
1355	15.72	.482	2.84	6.35	90	3.1	20.75
1400	15.72	.487	2.86	6.35	91	6.6	20.76
1405	15.72	.489	2.84	6.35	91	2.9	20.77
1410	15.71	.492	2.84	6.36	92	2.4	20.77
1415	15.71	.490	2.85	6.35	90	7.9	20.74
1420	15.70	.491	2.85	6.36	91	7.0	20.78

Sample collected @ 1420. Decommed
Supply. MOB to deliver sample cooler.
Fe < 2.2 Mn = 0.5



8/8/3
JH
SK

Arrived onsite @ 6:45. Had safety meeting
9:53. Calibrated hanta. set up at
54mw7. Well pump set @ 17.5'
Pump turned on @ 7:15. 300ml/m.

54mw07

Time	Temp	cond	DO	pH	orp	turb	Σ
7:20	12.90	.693	1.18	6.91	73	21.6	13.63
7:25	13.04	.587	2.39	6.80	81	17.4	13.65
7:30	13.03	.480	2.41	6.78	83	16.1	13.66
7:35	13.03	.478	2.42	6.75	84	16.8	13.66
7:40	13.04	.497	2.43	6.72	84	16.0	13.67
7:45	13.03	.477	2.41	6.71	85	15.2	13.67
7:50	13.05	.478	2.40	6.71	86	7.2	13.69

Samples collected @ 7:50.

Fe < .2 Mn < .05

Reconnected pump & mob to 54mw05.

Setup on 54mw05 @ 8:20.

Pump turned on @ 8:25. 300ml/m

54mw05

Time	Temp	Cond	DO	pH	orp	turb	Σ
8:30	14.44	.371	6.83	6.40	121	18.1	15.86
8:35	14.47	.350	6.27	6.33	125	5.8	15.87
8:40	14.51	.342	6.26	6.31	129	1.7	15.87
8:45	14.48	.335	6.26	6.30	129	0.2	15.88
8:50	14.48	.333	6.30	6.29	129	0.0	15.88

8/8/3
JH
SK

Time	Temp	Cond	DO	pH	orp	turb	Σ
8:55	14.48	.332	6.28	6.29	129	0.0	15.88
9:00	14.49	.332	6.29	6.30	129	0.0	15.89
9:05	15.49	.333	6.28	6.30	129	0.0	15.89

Sampled well @ 9:05. Fe < .2 Mn .05

Recon pump & mob to 54mw03.

Pump set @ 26'. Pump ran @ 250 ml/m.

54mw03

Time	Temp	Cond	DO	pH	orp	turb	Σ
9:50	14.41	.645	1.33	6.78	109	0.0	21.40
9:55	14.73	.643	1.26	6.79	103	0.0	21.43
10:00	15.07	.628	1.46	6.79	95	0.0	
10:05	15.08	.621	1.64	6.79	94	0.0	
10:10	15.08	.618	1.69	6.76	95	0.0	
10:15	15.08	.617	1.68	6.76	95	0.0	
10:20	15.08	.617	1.69	6.75	96	0.0	

Collected samples @ 10:20. Gave Jim McKenna
a demonstration of sampling procedures.

Fe < .2 Mn < .05

Recon pump & mob to 54mw02.

Well set @ 10:55 @ 26'.

Pump started @ 11:00. 250 ml/m.

54MW02

8
01

Time	temp	cond.	DO	pH	orp	turb	g'
1:05	15.31	.575	3.93	6.97	119	28.8	22.1
11:10	15.14	.595	1.03	6.93	105	7.1	
11:15	15.24	.595	0.84	6.92	92	1.2	21.1
11:20	15.23	.593	.78	6.92	89	0.0	21.1
11:25	15.20	.594	.79	6.92	84	0.0	21.1
11:30	15.25	.593	.80	6.91	83	0.0	21.1
11:35	15.25	.592	.80	6.91	84	0.0	

also collected.

Fe Mn =

Waste water was taken to the treatment plant. Pond from site

11.5.13
J. Hillebrand
M. Basits

12:40 Arrived @ Radford @ 7:00 for bodying. Held
12:45 Safety meeting (JSD & tailgate) while waiting for
12:50 Bodying. MOB to SUMV54 To meet w/
12:55 Matt Alberts for Army Safety meeting.
Calibrated the Horiba according to specs.
Readings passed the manufacturers specs.
Called dispatch to open up Gate 19E.
MOB to 54MW10. PID=0

$\nabla = 17.05$ well rate 22' @ 8:55

300 ml/min

Time	temp	cond	DO	pH	orp	turb	∇
9:00	13.96	.729	0.00	7.01	14	101	17.13
9:05	14.76	.659	2.43	6.97	3	63.7	17.15
9:10	15.10	.623	2.47	6.88	13	5.9	17.16
9:15	15.22	.618	2.45	6.91	16	0.6	17.17
9:20	15.26	.613	2.46	6.92	17	0.0	17.17
9:25	15.24	.614	2.45	6.89	18	0	17.18
9:30	15.25	.615	2.45	6.89	19	0	17.18
9:35	15.25	.615	2.45	6.89	18	0	17.18

sampled @ 9:35 Fe C.2 Mn C.05

Decommed pump & took Rinse blank RB 11513
@ 9:55 MOB To SUMV13.

Set up @ 54mmw13. well 21' @ 13'.

Pump turned on @ 10:45. 200ml/min → 21.1

Time	temp	cond	DO	pH	orp	turb	Fe
10:50	15.72	.645	1.36	7.37	43	64.2	21.14
10:55	15.94	.644	1.34	7.36	43	34.2	21.16
11:00	15.97	.644	1.33	7.36	43	12.1	21.16
11:05	15.96	.648	1.31	7.29	25	0.3	21.16
11:10	15.96	.647	1.32	7.29	26	0.4	21.16
11:15	15.96	.643	1.31	7.28	25	0.4	21.17
11:20	15.96	.643	1.32	7.29	25	0.4	21.17

Well stabilized @ 11:20. Samples collected @ 11:25. Fe = 4.2 Mn = .05.

Decon pump, mob to 54mmw02.

Set pump @ 27'. T 21.81'. pump turned on @ 12:05. 250ml/min.

54mmw2

Time	temp	cond	DO	pH	orp	turb	Fe
12:05	14.03	.634	2.89	7.45	116	6.2	21.86
12:10	14.04	.634	2.84	7.45	91	5.6	21.87
12:15	14.05	.635	2.81	7.45	86	0	" "
12:20	14.05	.634	2.76	7.45	92	0	" "
12:25	14.06	.634	2.74	7.45	84	0	" "
12:30	14.06	.634	2.74	7.45	89	0	21.88
12:35	14.06	.634	2.73	7.45	57	0	" "

54mmw2

Time	Temp	cond	DO	orp	turb	Fe
12:40	14.07	.634	2.71	53	0	21.71
12:45	14.08	.634	2.71	54	0	" "
12:50	14.08	.634	2.70	53	0	" "
12:55	14.09	.635	2.70	51	0	" "

Samples collected @ 1:00. Decon supplies mob to 54mmw12. Fe < .02 Mn < .05

Set well pump @ 28'. started pump @ 1:35.

54mmw12

Time	Temp	cond	DO	orp	turb	Fe
13:40	14.58	.558	1.31	89	448	25.98
13:45	14.84	.474	1.21	100	214	26.01
13:50	14.91	.470	1.19	112	13.0	26.04
13:55	14.98	.470	1.19	111	7.4	26.05
14:00	14.96	.470	1.18	105	3.7	26.05
14:05	14.96	.489	1.17	106	3.1	26.05
14:10	14.96	.469	1.17	107	2.0	26.06
14:15	14.96	.469	1.17	108	4.6	26.06

Stabilized @ 14:15. Fe < .2 Mn .05

Collected 54mmw12, 54mmw2 & ms/msO.

Decon. Dropped 15 gallon IOW @ Treatment plant. Left site for shipping of samples.

* have 2 bladder pumps.
Alternately Pumps.
i.e. 1 pump
sampling while 1 pump is deconned.

2.19.14

J. Hildebrand
B. Grimes

Arrived @ Guard Gate to get Budget
@ 7:00. Arrived onsite @ 8:00 for
safety meeting w/ Matt Alberts. JSD &
Tailgate focused on well, 1st needle
and pinch points. Called security to
get Gate 19E opened. ~~Arrived to 54mw10~~

54mw01 19.32

54mw10 16.00

54mw13 20.71

54mw12 25.51

Calibrated horiba according to spec checked at delay
while waiting for security to arrive, set
up on 54mw13.

54mw13

set @ 250ml/m
pump @ 21.5

Time	Temp	Cond	DO	pH	Orp	Turb	Fe
8:45	10.68	.545	3.68	7.04	151	271	26.74
8:50	11.67	.532	3.17	7.17	143	153	20.75
8:55	11.86	.517	2.64	7.20	141	103	20.74
9:00	11.99	.499	2.13	7.20	140	70.2	20.75
9:05	12.03	.491	2.04	7.20	140	59.1	20.76
9:10	12.04	.484	2.10	7.21	141	19.6	20.74
9:15	12.04	.482	2.10	7.21	141	10.4	20.75
9:20	12.04	.482	2.09	7.21	141	9.6	20.74
9:25	12.06	.481	2.09	7.21	142	14.1	20.74

2.19.14
SN/06

Time Temp Cond DO pH Orp Turb Fe
9:30 12.04 .481 2.09 7.21 142 14.0 20.74
well stabilized @ 9:30. Collected samples
and deconned pump. Pump was
used for rinse blank 54RBO219.14. Fe < 0.2
Mn < 0.5
Security arrived @ 9:50. MOB to 54mw10

Set up on 54mw10 @ 10:10. Pump set @ 21'
250ml/m

Time	Temp	Cond	DO	pH	Orp	Turb	Fe
10:15	11.27	.496	2.57	7.01	155	0.0	16.02
10:20	12.99	.557	1.40	6.83	156	0.0	16.05
10:25	13.19	.587	1.41	6.87	154	0.0	16.03
10:30	13.21	.587	1.43	6.89	153	0.0	16.03
10:35	13.20	.587	1.43	6.88	153	0.0	16.05
10:40	13.20	.587	1.43	6.89	153	0.0	16.05

Samples collected @ 10:40. Fe = 0.2 Mn = < 0.5

called gate guards to open 19E. Deconned
pump while waiting on gate guards.
MOB to 54mw01. Set up on well @ 11:10

Set up @ 29' 250ml/m.

54mw01

Time	Temp	Cond	DO	pH	Orp	Turb	Fe
11:15	11.44	.410	1.16	7.94	133	Ø	19.35
11:20	11.89	.413	1.62	8.06	131	Ø	19.36
11:25	12.31	.417	1.61	8.06	131	Ø	19.34
11:30	12.32	.417	1.60	8.09	130	Ø	19.36

54mw01

2.19.14
JH/BG

Time	Temp	Cond	DO	pH	orp	Turb	Σ
11:35	12.32	.417	1.45	8.09	128	Ø	19.34
11:40	12.33	.417	1.31	8.10	126	Ø	19.36
11:43	12.33	.417	1.31	8.10	126	Ø	19.35
11:50	12.34	.416	1.32	8.10	126	Ø	19.36
11:55	12.34	.416	1.32	8.10	127	Ø	19.36
12:00	12.34	.416	1.32	8.10	127	Ø	19.36

Well stabilized @ 12:00. Samples

collected @ 12:00. MS MW01

9 MS0 MW01 collected as well.

Fe = 4.2 Mn = 4.05.

MOB to 54mw12

54mw12

Sep pump @ 21'. 250 ml/min

Time	Temp	Cond	DO	pH	orp	Turb	Σ
12:55	14.50	.664	0.64	7.37	145	124	25.54
13:00	14.23	.646	0.69	7.20	146	106	25.55
13:05	14.15	.644	.72	7.21	147	68	25.53
13:10	14.11	.644	.71	7.20	149	41	25.53
13:15	14.12	.643	.70	7.20	150	15.4	25.53
13:20	14.12	.643	.70	7.20	150	9.8	25.53
13:25	14.12	.643	.70	7.21	150	3.2	25.54
13:30	14.11	.643	.70	7.21	150	3.8	25.53

Stable @ 1330. 54mw12 & 54tm12 collected.

Fe = 3.2 Mn = 4.05

2.19.14

Transported ~ 15 gal purge water to treatment plant. Demob from site.

8.28.14

8.28.14

C. H. Lebar

J. Schwaner

Arrived onsite @ 7:00 for loading.

Met w/ Safety officer for risk assessment
& work permit. Calculated PDI & Pst
according to Spec.

MOB to Symwio

▽ 16.71

Started @ 8:55

PDI = 0

well set @ 20'

300 ml/m

Time	Temp	Cond	DO	pH	orp	Turb	Fe
9:00	14.42	.688	2.82	7.33	124.2	5.8	16.73
9:05	12.91	.595	1.20	6.93	116.8	1.9	16.73
9:10	12.80	.590	1.31	6.89	117.5	3.0	16.73
9:15	12.78	.583	1.54	6.90	119.4	3.4	16.75
9:20	12.75	.579	1.57	6.91	122.5	3.7	16.73
9:25	12.79	.580	1.56	6.92	124.4	3.7	16.73
9:30	12.80	.582	1.56	6.93	126.1	3.9	16.73
9:35	12.81	.582	1.57	6.93	127.0	3.7	16.73
9:40	12.81	.582	1.56	6.93	127.4	3.8	16.75

Stable @ 9:40, well sampled

Sample ID: Symwio.

Fe ~~5.2~~ 5.2 MW = 0.10called Dispatch to gain entry to
to limited area.

MOB to Symwio.

▽ 20.63

PDI = 0

Set @ 39'

Started @ 10:35

Time	Temp	Cond	DO	pH	orp	Turb	Fe
10:40	17.34	.473	6.56	7.98	124.3	3.6	20.71
10:45	16.84	.470	3.21	7.92	140.7	3.8	20.72
10:50	16.63	.468	1.70	7.79	162.2	3.2	20.72
10:55	16.61	.467	1.68	7.77	171.8	3.0	20.72
11:00	16.63	.467	1.69	7.77	189.5	3.0	20.72
11:05	16.62	.469	1.67	7.77	191.9	3.2	20.73
11:10	16.61	.469	1.65	7.77	208.0	3.1	20.71
11:15	16.61	.469	1.65	7.79	216.8	3.1	20.73
11:20	16.61	.469	1.64	7.79	220.4	3.2	20.73
11:25	16.61	.469	1.65	7.79	222.3	3.0	20.71

Well stable @ 11:25 Symwio collected.

MS/mso collected Fe = 0.2 MW = 0.05

Decommed 2 well pumps & MOB to
Symwio 10. RB052814 collected @

10:25

5/28/14

MOB to 54mw12

7 24.5

84 @ 27.5

PID = 0

well started @

12:35

PN	Time	Temp	Cond	DO	orp	Turb	7	PN
7.32	12:40	16.63	.669	2.33	176.6	146.6	27.51	
7.33	12:45	15.39	.617	2.36	218.6	171.2	27.54	
7.34	12:50	14.78	.502	2.30	257.4	94.1	27.54	
7.34	12:55	14.63	.462	2.27	270.9	41.7	27.54	
7.34	13:00	14.67	.458	2.26	272.6	28.6	27.52	
7.34	13:05	14.65	.453	2.26	272.7	14.5	27.52	
7.34	13:10	14.61	.454	2.25	272.6	12.1	27.53	
7.34	13:15	14.61	.453	2.25	272.1	10.0	27.52	

Stable @ 13:15

Fe .2

Mn .10

collected 54mw12 759Tm12 @ 13:15

MOB to 54mw13

54 22.21
well set @ ~~24.75~~ 24.75 54
pump @ 2000 gpm
PID = 0
Started @ 14:10

Time	Temp	Cond	DO	orp	turb	7	PN
14:15	17.88	.549	1.21	-64.3	37.2	22.3	26.49
14:20	17.47	.526	1.17	-31.0	24.4	22.33	6.97
14:25	17.13	.514	1.16	-3.1	27.1	22.33	6.97
14:30	16.54	.483	1.16	49.4	16.2	22.34	6.88
14:35	16.59	.473	1.15	67.6	11.1	22.34	6.97
14:45	16.57	.471	1.15	69.7	8	22.34	6.97
14:50	16.57	.471	1.14	71.0	14	22.33	6.97
14:55	16.57	.470	1.14	71.3	9	22.33	6.97

well stable @ 14:55

54mw13. Fe = .02 Mn = .05

Demob from site disposed
of 20 gal purge @ treatment
plant.

Appendix B-1
Chain of
Custody
Forms



CHAIN-OF-CUSTODY RECORD

[illegible]



CHAIN-OF-CUSTODY RECORD

[illegible]



CHAIN-OF-CUSTODY RECORD

[illegible]

7334

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

~~TestAmerica Savannah~~
5102 LaRoche Avenue
Savannah, GA 31404

Website: www.testamericainc.com
Phone: (912) 354-7858
Fax: (912) 352-0165

○ Alternate Laboratory Name/Location

Phone:
Fax:

[illegible]

7333

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

[illegible]

Serial Number 83462

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

TestAmerica

TestAmerica Savannah
5102 LaRoche Avenue
Savannah, GA 31404

Website: www.testamericainc.com
Phone: (912) 354-7858
Fax: (912) 352-0165

THE LEADER IN ENVIRONMENTAL TESTING

Alternate Laboratory Name/Location

Phone:
Fax:

PROJECT REFERENCE		PROJECT NO.	PROJECT LOCATION (STATE)	MATRIX TYPE	REQUIRED ANALYSIS						PAGE	OF	
TAL (LAB) PROJECT MANAGER		P.O. NUMBER	CONTRACT NO.	COMPOSITE (C) OR GRAB (G) INDICATE	NONAQUEOUS LIQUID (OIL, SOLVENT, ...)	AIR	SOLID OR SEMISOLID	AQUEOUS (WATER)	STANDARD REPORT DELIVERY	DATE DUE	EXPEDITED REPORT DELIVERY (SURCHARGE)	DATE DUE	NUMBER OF COOLERS SUBMITTED PER SHIPMENT
Lisa Harvey		68011957	VIA						9060 DIC	9060 DIC	9060 DIC		
CLIENT (SITE) PM		410-273-7233	CLIENT FAX						9060 DIC	9060 DIC	9060 DIC		
CLIENT NAME		CBT-Encl. Malwarek	410-273-7100						9060 DIC	9060 DIC	9060 DIC		
CLIENT ADDRESS		Encl. Malwarek	Services, Inc.						9060 DIC	9060 DIC	9060 DIC		
COMPANY CONTRACTING THIS WORK (if applicable)		Millennium DC	Salcomp, Inc						9060 DIC	9060 DIC	9060 DIC		
SAMPLE		SAMPLE IDENTIFICATION		NUMBER OF CONTAINERS SUBMITTED									
DATE	TIME	DATE	TIME	3	6	9	3	9	3	3	3	3	3
5/28/14	1125	54mw01		X									
5/28/14	0940	54mw10		X									
5/28/14	1315	54mw12		X									
5/28/14	1455	54mw13		X									
5/28/14	1315	54mw12		X									
5/28/14	1725	54RB052814		X									



680-101795 Chain of Custody

RELINQUISHED BY: (SIGNATURE)		DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME
[Signature]		5/28/14	1700			
RECEIVED BY: (SIGNATURE)		DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME
[Signature]						

LABORATORY USE ONLY

RECEIVED FOR LABORATORY BY: (SIGNATURE)	DATE	TIME	CUSTODY INTACT YES <input type="radio"/> NO <input type="radio"/>	CUSTODY SEAL NO.	SAVANNAH LOG NO.	LABORATORY REMARKS
[Signature]	5/28/14	1000			680-101795	4.0/3.8°C

Serial Number

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

TestAmerica Savannah
5102 LaRoche Avenue
Savannah, GA 31404

Website: www.testamericainc.com
Phone: (912) 354-7858
Fax: (912) 352-0165

TestAmerica

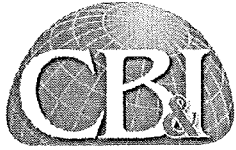
THE LEADER IN ENVIRONMENTAL TESTING

○	Alternate Laboratory Name/Location
---	------------------------------------

Phone: _____
Fax: _____

PROJECT REFERENCE	PROJECT NO. Lab	PROJECT LOCATION (STATE) VA	PROJECT NO.	MATRIX TYPE	REQUIRED ANALYSIS	PAGE 1	OF 1
TAL (LAB) PROJECT MANAGER	P.O. NUMBER	CLIENT PHONE	CLIENT FAX	COMPOSITE (C) OR GRAB (G) INDICATE	NONAQUEOUS LIQUID (OIL, SOLVENT, ...)	STANDARD REPORT DELIVERY	
CLIENT (SITE) PM	CLIENT E-MAIL	CLIENT ADDRESS	COMPANY CONTRACTING THIS WORK (if applicable)	AIR	9060-DIC	DATE DUE	
CLIENT NAME	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	SOLID OR SEMISOLID	9060-DIC	EXPEDITED REPORT DELIVERY (SURCHARGE)	
CLIENT ADDRESS	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	AQUEOUS (WATER)	9060-DIC	DATE DUE	
COMPANY CONTRACTING THIS WORK (if applicable)	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	COMPOSITE (C) OR GRAB (G) INDICATE	9060-DIC	NUMBER OF COOLERS SUBMITTED PER SHIPMENT:	
PROJECT REFERENCE	PROJECT NO. Lab	PROJECT LOCATION (STATE) VA	PROJECT NO.	MATRIX TYPE	REQUIRED ANALYSIS	PAGE 1	OF 1
TAL (LAB) PROJECT MANAGER	P.O. NUMBER	CLIENT PHONE	CLIENT FAX	COMPOSITE (C) OR GRAB (G) INDICATE	NONAQUEOUS LIQUID (OIL, SOLVENT, ...)	STANDARD REPORT DELIVERY	
CLIENT (SITE) PM	CLIENT E-MAIL	CLIENT ADDRESS	COMPANY CONTRACTING THIS WORK (if applicable)	AIR	9060-DIC	DATE DUE	
CLIENT NAME	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	SOLID OR SEMISOLID	9060-DIC	EXPEDITED REPORT DELIVERY (SURCHARGE)	
CLIENT ADDRESS	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	AQUEOUS (WATER)	9060-DIC	DATE DUE	
COMPANY CONTRACTING THIS WORK (if applicable)	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	COMPOSITE (C) OR GRAB (G) INDICATE	9060-DIC	NUMBER OF COOLERS SUBMITTED PER SHIPMENT:	
PROJECT REFERENCE	PROJECT NO. Lab	PROJECT LOCATION (STATE) VA	PROJECT NO.	MATRIX TYPE	REQUIRED ANALYSIS	PAGE 1	OF 1
TAL (LAB) PROJECT MANAGER	P.O. NUMBER	CLIENT PHONE	CLIENT FAX	COMPOSITE (C) OR GRAB (G) INDICATE	NONAQUEOUS LIQUID (OIL, SOLVENT, ...)	STANDARD REPORT DELIVERY	
CLIENT (SITE) PM	CLIENT E-MAIL	CLIENT ADDRESS	COMPANY CONTRACTING THIS WORK (if applicable)	AIR	9060-DIC	DATE DUE	
CLIENT NAME	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	SOLID OR SEMISOLID	9060-DIC	EXPEDITED REPORT DELIVERY (SURCHARGE)	
CLIENT ADDRESS	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	AQUEOUS (WATER)	9060-DIC	DATE DUE	
COMPANY CONTRACTING THIS WORK (if applicable)	CLIENT PHONE	CLIENT FAX	CLIENT E-MAIL	COMPOSITE (C) OR GRAB (G) INDICATE	9060-DIC	NUMBER OF COOLERS SUBMITTED PER SHIPMENT:	

Appendix B-2
Data
Validation



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Belcamp, Maryland 21017
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Fax: +1 (410) 273-7103
Eric.malarek@CBIfederalservices.com

MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon
Microbac Laboratories, Inc. L13080490

DATE: March 18, 2014

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, 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 eight aqueous samples were validated. The sample IDs are:


Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW07	L13080490-01	54MW03	L13080490-05
54MW07	L13080490-02	54MW03	L13080490-06
54MW05	L13080490-03	54MW02	L13080490-07
54MW05	L13080490-04	54MW02	L13080490-08

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

3/18/14

 Date

**RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13080490**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, 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 08/08/13, the coolers were received on 08/09/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 2.0°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: The samples were collected on 08/08/13. The TOC and TIC analysis were run on 08/13/13. The DOC and DIC analysis were run on 08/13/13. 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 08/13/13. The DOC and DIC analysis were run on 08/13/13. The initial calibration for TC was analyzed on 07/09/13 with a coefficient of determination of 0.9996. The initial calibration for TIC was analyzed on 07/09/13 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 54MW07 (L13080490-01), 54MW07 (L13080490-02), 54MW05 (L13080490-03), 54MW05 (L13080490-04), 54MW03 (L13080490-05), 54MW03 (L13080490-06), 54MW02 (L13080490-07), and 54MW02 (L13080490-08) 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 ½MRL (<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 54RB080713 (L13080428-09) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB080713 (L13080428-09) 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)
08/12/13	TOC	ICB/CCBs	<LOD	NA	None
08/13/13	TOC	ICB/CCBs	<LOD	NA	None
08/12/13	TOC	WG440968-01	<½MRL	NA	None
08/13/13	TOC	WG440969-01	<½MRL	NA	None
08/13/13	DOC	WG440969-01	<½MRL	NA	None
08/12/13	TOC	54RB080713	0.607J	3.04	54MW07, 54MW05, 54MW03, 54MW02
08/12/13	TIC	54RB080713	<½MRL	NA	None
08/13/13	DOC	54RB080713	1.45J	7.25	54MW07, 54MW05, 54MW03, 54MW02
08/13/13	DIC	54RB080713	<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 WG440968-02 and WG440968-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 08/12/13. All criteria were met. No qualifiers were applied. Samples 54MW07 (L13080490-01) and 54MW05 (L13080490-03) apply to this LCS/LCSD.
- Samples WG440969-02 and WG440969-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 08/13/13. All criteria were met. No qualifiers were applied. Samples 54MW03 (L13080490-05) and 54MW02 (L13080490-07) apply to this LCS/LCSD.
- Samples WG440969-02 and WG440969-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 08/13/13. All criteria were met. No qualifiers were applied. Samples 54MW07 (L13080490-02), 54MW05 (L13080490-04), 54MW03 (L13080490-06), and 54MW02 (L13080490-08) 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%).

- Sample 54MW02 (L13080490-07) was used as the aqueous MS/MSD for TOC and TIC analysis on 08/13/13. All criteria were met. No qualifiers were applied. Samples 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) apply to this MS/MSD.
- Sample 54MW02 (L13080490-08) was used as the aqueous MS/MSD for DOC and DIC analysis on 08/13/13. DOC (79.6%) was outside criteria. The parent spiked sample was qualified "L" bias low based upon the low recovery. Samples 54MW07 (L13080490-02), 54MW05 (L13080490-04), 54MW03 (L13080490-06), and 54MW02 (L13080490-08) 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 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 54MW07 (L13080490-01), 54MW07 (L13080490-02), 54MW05 (L13080490-03), 54MW05 (L13080490-04), 54MW03 (L13080490-05), 54MW03 (L13080490-06), 54MW02 (L13080490-07), and 54MW02 (L13080490-08) 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
54MW07	TOC/DOC	1.0; 2.0	0.822J	2.73	69.9	J
54MW07	TIC/DIC	1.0; 2.0	0.825J	17.9	95.4	J
54MW05	TOC/DOC	1.0; 2.0	0.908J	5.35	83.0	J
54MW05	TIC/DIC	1.0; 2.0	1.75	36.0	95.1	J
54MW03	TOC/DOC	1.0; 2.0	1.11	5.77	80.8	J
54MW03	TIC/DIC	1.0; 2.0	0.597J	53.1	98.9	J
54MW02	TOC/DOC	1.0; 2.0	1.50	4.95	69.7	J
54MW02	TIC/DIC	1.0; 2.0	0.735J	42.1	98.3	J

J = Estimated value.

MRL = Method Reporting Limit

NA = Not Applicable

Sample: 54MW07 (L13080490-01), TOC

$$TC: Y = m \cdot X \text{ (mg/L)} + b$$

$$m = 36.20$$

$$b = 6.332$$

$$Y = 65.97$$

$$DF = 1$$

$$TIC: Y = m \cdot X \text{ (mg/L)} + b$$

$$m = 29.78$$

$$b = 8.713$$

$$Y = 33.29$$

$$DF = 1$$

$$X = (1.65 \text{ mg/L}) \cdot 1 = 1.65 \text{ mg/L}$$

$$X = (0.8253 \text{ mg/L}) \cdot 1 = 0.8253 \text{ mg/L}$$

$$TOC \text{ (mg/L)} = TC \text{ (mg/L)} - TIC \text{ (mg/L)} = 1.65 - 0.8253 = 0.824 \text{ mg/L}$$

$$\text{Reported Value} = 0.822 \text{ mg/L}$$

$$\% \text{ Difference} = 0.24\%$$

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Microbac

Lab Report #: L13080490
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-01	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/13/2013 13:01
Collect Date: 08/08/2013 07:50	Dilution: 1	File ID: TC08122013.030
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.822	J B	1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Microbac

Lab Report #: L13080490
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-01	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/13/2013 13:01
Collect Date: 08/08/2013 07:50	Dilution: 1	File ID: TC08122013.030
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		0.825	J J	1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Microbac

Lab Report #: L13080490
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-02	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:15
Collect Date: 08/08/2013 07:50	Dilution: 2	File ID: TC08122013.045
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	2.73	B	2.00	1.00

Sample #: L13080490-02	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:15
Collect Date: 08/08/2013 07:50	Dilution: 2	File ID: TC08122013.045
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		17.9	J	2.00	1.00

Sample #: L13080490-03	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/13/2013 13:13
Collect Date: 08/08/2013 09:05	Dilution: 1	File ID: TC08122013.031
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		1.75	J	1.00	0.500

Sample #: L13080490-03	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/13/2013 13:13
Collect Date: 08/08/2013 09:05	Dilution: 1	File ID: TC08122013.031
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.908	J B	1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Microbac

Lab Report #: L13080490
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-04	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:28
Collect Date: 08/08/2013 09:05	Dilution: 2	File ID: TC08122013.046
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		36.0	J	2.00	1.00

Sample #: L13080490-04	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:28
Collect Date: 08/08/2013 09:05	Dilution: 2	File ID: TC08122013.046
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	5.35	B	2.00	1.00

Sample #: L13080490-05	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 18:09
Collect Date: 08/08/2013 10:20	Dilution: 1	File ID: TC08122013.054
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.11	B	1.00	0.500

Sample #: L13080490-05	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 18:09
Collect Date: 08/08/2013 10:20	Dilution: 1	File ID: TC08122013.054
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		0.597	J J	1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Microbac

Lab Report #: L13080490
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-06	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:42
Collect Date: 08/08/2013 10:20	Dilution: 2	File ID: TC08122013.047
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	5.77	B	2.00	1.00

Sample #: L13080490-06	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:42
Collect Date: 08/08/2013 10:20	Dilution: 2	File ID: TC08122013.047
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		53.1	J	2.00	1.00

Sample #: L13080490-07	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 18:21
Collect Date: 08/08/2013 11:35	Dilution: 1	File ID: TC08122013.055
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	1.50	B	1.00	0.500

Sample #: L13080490-07	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 18:21
Collect Date: 08/08/2013 11:35	Dilution: 1	File ID: TC08122013.055
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		0.735	J	1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Microbac

Lab Report #: L13080490

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-08	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:55
Collect Date: 08/08/2013 11:35	Dilution: 2	File ID: TC08122013.048
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		42.1	J	2.00	1.00

Sample #: L13080490-08	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:55
Collect Date: 08/08/2013 11:35	Dilution: 2	File ID: TC08122013.048
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.95	B	2.00	1.00

Sample #: L13080490-08	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 18:33
Collect Date: 08/08/2013 11:35	Dilution: 1	File ID: TC08122013.056
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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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13080490

DATE: March 18, 2014

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, 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
54MW07	L13080490-01	54MW03	L13080490-05
54MW05	L13080490-03	54MW02	L13080490-07

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

3/18/14
 Date

**RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13080490**

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 08/08/13, the coolers were received on 08/09/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 2.0°C. All criteria were met. No qualifiers were applied.
- Holding Time Review: The aqueous samples were collected on 08/08/13 for perchlorate analysis. The aqueous samples were prepped and analyzed on 08/14/13. 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, ¹⁸O-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 $RSD \leq 20\%$ DoD Perchlorate Handbook)
 ICV ($\leq 15\%D$ DoD Perchlorate Handbook)
 CCV/ICS ($\leq 15\%D$ DoD Perchlorate Handbook)
 LODV ($\pm 30\%D$ DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 07/12/13 on instrument LCMS1, all criteria were met for all target compounds ($RSD \leq 20\%$). 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 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) apply to this initial and continuing calibrations.

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 54RB080713 (L13080428-09) 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)
08/14/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
08/14/13	WG441352-02	All perchlorate <½MRL	NA	NA	None
08/13/13	54RB080713	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) $1.0 \pm 2\%$ 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 WG441352-03 was used as aqueous LCS for perchlorate analysis dated 08/14/13. All criteria were met. No qualifiers were applied. Samples 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) 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%.

- Sample 54MW02 (L13080490-07) was used as aqueous MS/MSD for perchlorate analysis dated 08/14/13. Perchlorate (70%) was below criteria. The parent spiked sample was qualified "L" bias low based upon the low recovery. The RPD for the MS/MSD was within criteria limits. Samples 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) 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.

- 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 (L13080490-07), 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:

$$m = 1.37$$

$$b = 0.00457$$

$$Y = \text{Area} = 97300/350000 = 0.278$$

$$X = 0.1996$$

$$\text{Conc. } \mu\text{g/L} = (A_x * C_{is} * DF)$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

A_x = Amount Ratio = Conc. Analyte/Conc. Internal Standard

C_{is} = Conc. Of internal Standard ($\mu\text{g/L}$)

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (0.1996 * 5 * 1) = 1.00 \mu\text{g/L (Signal \#1)}$$

Reported Value = 1.00 $\mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Microbac

Lab Report #: L13080490

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-01	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW07	Prep Method: 6850	Prep Date: 08/14/2013 13:30
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441352	Analyst: JWR	Run Date: 08/14/2013 16:39
Collect Date: 08/08/2013 07:50	Dilution: 1	File ID: 1LM.LM22111
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.370		0.200	0.100

Sample #: L13080490-03	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW05	Prep Method: 6850	Prep Date: 08/14/2013 13:30
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441352	Analyst: JWR	Run Date: 08/14/2013 16:58
Collect Date: 08/08/2013 09:05	Dilution: 1	File ID: 1LM.LM22112
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.389		0.200	0.100

Sample #: L13080490-05	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW03	Prep Method: 6850	Prep Date: 08/14/2013 13:30
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441352	Analyst: JWR	Run Date: 08/14/2013 17:17
Collect Date: 08/08/2013 10:20	Dilution: 1	File ID: 1LM.LM22113
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.446		0.200	0.100

Sample #: L13080490-07	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW02	Prep Method: 6850	Prep Date: 08/14/2013 13:30
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441352	Analyst: JWR	Run Date: 08/14/2013 15:42
Collect Date: 08/08/2013 11:35	Dilution: 1	File ID: 1LM.LM22108
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	1.00	L	0.200	0.100



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13080490

DATE: March 18, 2014

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, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of four aqueous samples were validated. The sample IDs are:


Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW07	L13080490-01	54MW03	L13080490-05
54MW05	L13080490-03	54MW02	L13080490-07

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

3/18/14
 Date

**RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13080490**

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 ($@4^{\circ}\text{C} \pm 2^{\circ}\text{C}$) 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 08/08/13, the coolers were received on 08/09/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 2.0°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: The aqueous samples were collected on 08/08/13. The samples were extracted on 08/14/13 and were analyzed on 08/15/13 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 54RB080713 (L13080428-09) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. $\mu\text{g/L}$	Action Level $\mu\text{g/L}$	B qualified samples (For this SDG)
08/15/13	WG441278-01	All target explosives $< \frac{1}{2}\text{MRL}$	NA	NA	None
08/15/13	54RB080713	All target explosives $< \frac{1}{2}\text{MRL}$	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 $\leq 20\%$.

- 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 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) 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 $\pm 20\%$.

- 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 field samples were reported using this initial verification calibration.
- For explosives continuing calibration performed on 08/14/13 @23:07 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous field samples were reported using this continuing calibration.
- For explosives continuing calibration performed on 08/15/13 @07:14 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous field samples were reported using this continuing calibration.
- For explosives continuing calibration performed on 08/15/13 @19:17 on instrument HPLC5, all criteria were met. No qualifiers were applied. Field samples 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) were reported using this continuing calibration.
- For explosives continuing calibration performed on 08/16/13 @01:26 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous field 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

Laboratory control samples (LCS) 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.

- Sample WG441278-02 was used as the aqueous LCS for the explosives analysis on 08/15/13. All criteria were met. No qualifiers were applied. Samples 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) 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 $\leq 20\%$ RPD.

- Sample 54MW02 (L13080490-07) was used as the aqueous MS/MSD for the explosives analysis on 08/15/13. Target compound 4-nitrotoluene (200%; RPD=74.4%) was outside criteria. For all other target compounds, all criteria were met. Target compound 4-nitrotoluene was non-detect for the spiked sample; therefore, was qualified estimated "UJ" based upon the high RPD. Samples 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) 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 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).
- Any positive detections were confirmed by mass spectrum (MS) analysis. All MS confirmations were within criteria for all detected target analytes in this SDG. No qualifiers were applied.

Sample: 54MW02 (L13080490-07), 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 = 23.70696

$$X = 88.27$$

$$\text{Conc. } \mu\text{g/L} = (A_x * V_t * DF) / (V_s)$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

A_x = Amount of compound being measured ($\mu\text{g/L}$).

V_t = Volume of total extract (mL) from bench sheet.

V_s = Volume of sample extracted (mL).

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (491.30 * 10 * 1) / (900) = 0.981 \mu\text{g/L (Signal \#1)}$$

Reported Value = 0.981 $\mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Microbac

Lab Report #: L13080490
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-01	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54MW07	Prep Method: METHOD	Prep Date: 08/14/2013 08:00
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441348	Analyst: JWR	Run Date: 08/15/2013 19:56
Collect Date: 08/08/2013 07:50	Dilution: 1	File ID: 5L010821.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	92.5	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

Microbac

Lab Report #: L13080490
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-03
 Client ID: 54MW05
 Matrix: Water
 Workgroup #: WG441348
 Collect Date: 08/08/2013 09:05
 Sample Tag: 01

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

Instrument: HPLC5
 Prep Date: 08/14/2013 08:00
 Cal Date: 03/20/2013 17:01
 Run Date: 08/15/2013 20:35
 File ID: 5L010822.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.20	0.301
2,4-Dinitrotoluene	121-14-2		U	1.20	0.301
2,6-Dinitrotoluene	606-20-2		U	1.20	0.301
2,4,6-Trinitrotoluene	118-96-7		U	1.20	0.301
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.20	0.301
2-Nitrotoluene	88-72-2		U	1.20	0.301
4-Nitrotoluene	99-99-0		U	1.20	0.301
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.20	0.301
RDX	121-82-4		U	1.20	0.301
Nitroglycerin	55-63-0		U	1.20	0.301
Surrogate	Recovery	Lower Limit	Upper Limit	Q	
1,2-Dinitrobenzene	89.5	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

Microbac

Lab Report #: L13080490

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-05

PrePrep Method: N/A

Instrument: HPLC5

Client ID: 54MW03

Prep Method: METHOD

Prep Date: 08/14/2013 08:00

Matrix: Water

Analytical Method: 8330B

Cal Date: 03/20/2013 17:01

Workgroup #: WG441348

Analyst: JWR

Run Date: 08/15/2013 21:14

Collect Date: 08/08/2013 10:20

Dilution: 1

File ID: 5L010823.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	95.5	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

Microbac

Lab Report #: L13080490

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-07	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54MW02	Prep Method: METHOD	Prep Date: 08/14/2013 08:00
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441348	Analyst: JWR	Run Date: 08/15/2013 21:53
Collect Date: 08/08/2013 11:35	Dilution: 1	File ID: 5L010824.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	0.974	J J	1.11	0.278
2-Amino-4,6-dinitrotoluene	35572-78-2	1.53		1.11	0.278
2-Nitrotoluene	88-72-2		U	1.11	0.278
4-Nitrotoluene	99-99-0		U UJ	1.11	0.278
4-Amino-2,6-dinitrotoluene	19406-51-0	0.981	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	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, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13080490

DATE: March 18, 2014

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, 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
54MW07	L13080490-01	54MW03	L13080490-05
54MW05	L13080490-03	54MW02	L13080490-07

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.

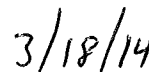
Table 1 Laboratory Performance Criteria

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



Date

**RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13080490**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 28 days for sulfate and chloride and Cool to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ with H_2SO_4 to $\text{pH} < 2$ 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 08/08/13, the coolers were received on 08/09/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 2.0°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: Samples were collected on 08/08/13. The samples were prepped and analyzed on 08/09/13 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 07/23/13 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 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) 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 54RB080713 (L13080428-09) 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)
08/09/13	Chloride	ICB/CCBs	<LOD	NA	None
08/09/13	Sulfate	ICB/CCBs	<LOD	NA	None
08/09/13	Nitrate	ICB/CCBs	<LOD	NA	None
08/09/13	Chloride	WG440828-01	<½MRL	NA	None
08/09/13	Sulfate	WG440828-01	<½MRL	NA	None
08/09/13	Nitrate	WG440828-01	<½MRL	NA	None
08/09/13	Chloride	54RB080713	0.117	0.585	None
08/09/13	Sulfate	54RB080713	<½MRL	NA	None
08/09/13	Nitrate	54RB080713	<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 WG440828-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 08/09/13. All criteria were met. No qualifiers were applied. Samples 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) 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 \leq 20% (DOD QSM = 80-120%; RPD \leq 20%)

- Sample 54MW02 (L13080490-07) was used as the aqueous MS/MSD for chloride, sulfate, and nitrate analysis on 08/09/13. All criteria were met. No qualifiers were applied. Samples 54MW07 (L13080490-01), 54MW05 (L13080490-03), 54MW03 (L13080490-05), and 54MW02 (L13080490-07) 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 analysis 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: 54MW07 (L13080490-01), sulfate

$$Y = mX + b$$

Y = Sample Area

m = slope of curve

X = Concentration (mg/L)

b = Y-intercept

DF = Dilution Factor

Given:

$$m = 0.1355$$

$$b = -0.0194$$

$$Y = \text{Area} = 3.677$$

$$DF = 1$$

$$X = 27.3 \text{ mg/L} * DF = 27.3 \text{ mg/L} * 1 = 27.3 \text{ mg/L}$$

$$\text{Reported concentration} = 27.3 \text{ mg/L}$$

$$\%D = 0.0\%$$

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Microbac

Lab Report #: L13080490
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-01	PrePrep Method: N/A	Instrument: IC2
Client ID: 54MW07	Prep Method: 9056	Prep Date: 08/09/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 07/23/2013 18:03
Workgroup #: WG440828	Analyst: KRB	Run Date: 08/09/2013 15:36
Collect Date: 08/08/2013 07:50	Dilution: 1	File ID: I2_080913-07
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	2.35		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	27.3		1.00	0.500
U	Analyte was not detected. The concentration is below the reported LOD.				

Microbac

Lab Report #: L13080490

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-03	PrePrep Method: N/A	Instrument: IC2
Client ID: 54MW05	Prep Method: 9056	Prep Date: 08/09/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 07/23/2013 18:03
Workgroup #: WG440828	Analyst: KRB	Run Date: 08/09/2013 15:55
Collect Date: 08/08/2013 09:05	Dilution: 1	File ID: I2_080913-08
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	7.83		0.200	0.100
Nitrate	14797-55-8	0.849		0.600	0.100
Sulfate	14808-79-8	15.0		1.00	0.500

Sample #: L13080490-05	PrePrep Method: N/A	Instrument: IC2
Client ID: 54MW03	Prep Method: 9056	Prep Date: 08/09/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 07/23/2013 18:03
Workgroup #: WG440828	Analyst: KRB	Run Date: 08/09/2013 16:13
Collect Date: 08/08/2013 10:20	Dilution: 1	File ID: I2_080913-09
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.84		0.200	0.100
Nitrate	14797-55-8	0.521	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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Lab Report #: L13080490
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080490-07	PrePrep Method: N/A	Instrument: IC2
Client ID: 54MW02	Prep Method: 9056	Prep Date: 08/09/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 07/23/2013 18:03
Workgroup #: WG440828	Analyst: KRB	Run Date: 08/09/2013 16:32
Collect Date: 08/08/2013 11:35	Dilution: 1	File ID: I2_080913-10
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	6.06		0.200	0.100
Nitrate	14797-55-8	0.326	J J	0.600	0.100
Sulfate	14808-79-8	30.8		1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon
Microbac Laboratories, Inc. L13080428

DATE: January 13, 2014

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, 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
54MW09	L13080428-01	54MW10	L13080428-07
54MW09	L13080428-02	54MW10	L13080428-08
54MW11	L13080428-03	54RB080713	L13080428-09
54MW11	L13080428-04	54RB080713	L13080428-10
54MW08	L13080428-05	54MW12	L13080428-11
54MW08	L13080428-06	54MW12	L13080428-12

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

1/13/14

 Date

**RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13080428**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, 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 08/07/13, the coolers were received on 08/08/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C and 3.0°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: The samples were collected on 08/07/13. The TOC and TIC analysis were run on 08/12/13 and 08/13/13. The DOC and DIC analysis were run on 08/13/13. 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 08/12/13 and 08/13/13. The DOC and DIC analysis were run on 08/13/13. The initial calibration for TC was analyzed on 07/09/13 with a coefficient of determination of 0.9996. The initial calibration for TIC was analyzed on 07/09/13 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 (L13080428-01), 54MW09 (L13080428-02), 54MW11 (L13080428-03), 54MW11 (L13080428-04), 54MW08 (L13080428-05), 54MW08 (L13080428-06), 54MW10 (L13080428-07), 54MW10 (L13080428-08), 54RB080713 (L13080428-09), 54RB080713 (L13080428-10), 54MW12 (L13080428-11), and 54MW12 (L13080428-12) 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 ½MRL (<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 54RB080713 (L13080428-09) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB080713 (L13080428-09) 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)
08/12/13	TOC	ICB/CCBs	<LOD	NA	None
08/13/13	TOC	ICB/CCBs	<LOD	NA	None
08/12/13	TOC	WG440968-01	<½MRL	NA	None
08/13/13	DOC	WG440969-01	<½MRL	NA	None
08/12/13	TOC	54RB080713	0.607J	3.04	54MW08
08/12/13	TIC	54RB080713	<½MRL	NA	None
08/13/13	DOC	54RB080713	1.45J	7.25	54MW09, 54MW11, 54MW08, 54MW10, 54MW12
08/13/13	DIC	54RB080713	<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 WG440968-02 and WG440968-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 08/12/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), and 54MW12 (L13080428-11) apply to this LCS/LCSD.
- Samples WG440969-02 and WG440969-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 08/13/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13080428-02), 54MW11 (L13080428-04), 54MW08 (L13080428-06), 54MW10 (L13080428-08), 54RB080713 (L13080428-10), and 54MW12 (L13080428-12) 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 site specific aqueous MS/MSD was performed for TOC and TIC analysis on 08/07/13; 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 08/08/13; 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 54MW09 (L13080428-01), 54MW09 (L13080428-02), 54MW11 (L13080428-03), 54MW11 (L13080428-04), 54MW08 (L13080428-05), 54MW08 (L13080428-06), 54MW10 (L13080428-07), 54MW10 (L13080428-08), 54RB080713 (L13080428-09), 54RB080713 (L13080428-10), 54MW12 (L13080428-11), and 54MW12 (L13080428-12) 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	1.0; 2.0	4.17	5.04	17.3	J
54MW09	TIC/DIC	1.0; 2.0	18.4	38.0	51.6	J
54MW11	TOC/DOC	1.0; 2.0	3.11	5.73	45.7	J
54MW11	TIC/DIC	1.0; 2.0	37.6	59.9	37.2	J
54MW08	TOC/DOC	1.0; 2.0	2.30	4.89	53.0	J
54MW08	TIC/DIC	1.0; 2.0	36.5	58.0	37.1	J
54MW10	TOC/DOC	1.0; 2.0	4.28	5.74	25.4	J
54MW10	TIC/DIC	1.0; 2.0	39.3	51.4	23.5	J
54RB080713	TOC/DOC	1.0; 2.0	0.607J	1.45	58.2	J
54RB080713	TIC/DIC	1.0; 2.0	1.00U	2.00U	NA	None
54MW12	TOC/DOC	1.0; 2.0	3.27	5.71	42.7	J
54MW12	TIC/DIC	1.0; 2.0	3.74	48.1	92.2	J

J = Estimated value.

MRL = Method Reporting Limit

NA = Not Applicable

Sample: 54MW09 (L13080428-01), TOC

TC: $Y = m \cdot X \text{ (mg/L)} + b$

$m = 36.20$

$b = 6.332$

$Y = 823.5$

$DF = 1$

TIC: $Y = m \cdot X \text{ (mg/L)} + b$

$m = 29.78$

$b = 8.713$

$Y = 556.7$

$DF = 1$

$X = (22.57 \text{ mg/L}) \cdot 1 = 22.57 \text{ mg/L}$

$X = (18.40 \text{ mg/L}) \cdot 1 = 18.40 \text{ mg/L}$

$\text{TOC (mg/L)} = \text{TC (mg/L)} - \text{TIC (mg/L)} = 22.57 - 18.40 = 4.17 \text{ mg/L}$

Reported Value = 4.17 mg/L

% Difference = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

²The 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 #: L13080428
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-01	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/12/2013 17:59
Collect Date: 08/07/2013 09:50	Dilution: 1	File ID: TC08122013.006
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	4.17	J	1.00	0.500

Sample #: L13080428-01	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/12/2013 17:59
Collect Date: 08/07/2013 09:50	Dilution: 1	File ID: TC08122013.006
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		18.4	J	1.00	0.500

Sample #: L13080428-02	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 14:23
Collect Date: 08/07/2013 09:50	Dilution: 2	File ID: TC08122013.037
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		38.0	J	2.00	1.00

Sample #: L13080428-02	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 14:23
Collect Date: 08/07/2013 09:50	Dilution: 2	File ID: TC08122013.037
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	5.04	B	2.00	1.00

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Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-03	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/12/2013 18:12
Collect Date: 08/07/2013 10:55	Dilution: 1	File ID: TC08122013.007
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.11	J	1.00	0.500

Sample #: L13080428-03	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/12/2013 18:12
Collect Date: 08/07/2013 10:55	Dilution: 1	File ID: TC08122013.007
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		37.6	J	1.00	0.500

Sample #: L13080428-04	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 14:37
Collect Date: 08/07/2013 10:55	Dilution: 2	File ID: TC08122013.038
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		59.9	J	2.00	1.00

Sample #: L13080428-04	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 14:37
Collect Date: 08/07/2013 10:55	Dilution: 2	File ID: TC08122013.038
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	5.73	B	2.00	1.00

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Lab Report #: L13080428
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-05	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/12/2013 18:26
Collect Date: 08/07/2013 12:05	Dilution: 1	File ID: TC08122013.008
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.30	B	1.00	0.500

Sample #: L13080428-05	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/12/2013 18:26
Collect Date: 08/07/2013 12:05	Dilution: 1	File ID: TC08122013.008
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		36.5	J	1.00	0.500

Sample #: L13080428-06	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 14:51
Collect Date: 08/07/2013 12:05	Dilution: 2	File ID: TC08122013.039
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.89	B	2.00	1.00

Sample #: L13080428-06	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 14:51
Collect Date: 08/07/2013 12:05	Dilution: 2	File ID: TC08122013.039
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		58.0	J	2.00	1.00

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Lab Report #: L13080428
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-07	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/12/2013 18:41
Collect Date: 08/07/2013 13:15	Dilution: 1	File ID: TC08122013.009
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		39.3	J	1.00	0.500

Sample #: L13080428-07	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/12/2013 18:41
Collect Date: 08/07/2013 13:15	Dilution: 1	File ID: TC08122013.009
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	4.28	J	1.00	0.500

Sample #: L13080428-08	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 15:38
Collect Date: 08/07/2013 13:15	Dilution: 2	File ID: TC08122013.042
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	5.74	B	2.00	1.00

Sample #: L13080428-08	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 15:38
Collect Date: 08/07/2013 13:15	Dilution: 2	File ID: TC08122013.042
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		51.4	J	2.00	1.00

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Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13080428-09	PrePrep Method:	N/A	Instrument:	TOC-VWP
Client ID:	54RB080713	Prep Method:	415.1	Prep Date:	N/A
Matrix:	Water	Analytical Method:	415.1	Cal Date:	07/09/2013 14:51
Workgroup #:	WG440968	Analyst:	DIH	Run Date:	08/12/2013 18:54
Collect Date:	08/07/2013 13:35	Dilution:	1	File ID:	TC08122013.010
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 #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13080428-09	PrePrep Method:	N/A	Instrument:	TOC-VWP
Client ID:	54RB080713	Prep Method:	415.1	Prep Date:	N/A
Matrix:	Water	Analytical Method:	415.1	Cal Date:	07/09/2013 14:51
Workgroup #:	WG440968	Analyst:	DIH	Run Date:	08/12/2013 18:54
Collect Date:	08/07/2013 13:35	Dilution:	1	File ID:	TC08122013.010
Sample Tag:	01	Units:	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	0.607	J J	1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

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Lab Report #: L13080428
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-10	PrePrep Method: N/A	Instrument: TOC-VWP
Client ID: 54RB080713	Prep Method: 415.1	Prep Date: N/A
Matrix: Water	Analytical Method: 415.1	Cal Date: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 15:50
Collect Date: 08/07/2013 13:35	Dilution: 2	File ID: TC08122013.043
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 #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-10	PrePrep Method: N/A	Instrument: TOC-VWP
Client ID: 54RB080713	Prep Method: 415.1	Prep Date: N/A
Matrix: Water	Analytical Method: 415.1	Cal Date: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 15:50
Collect Date: 08/07/2013 13:35	Dilution: 2	File ID: TC08122013.043
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	1.45	J J	2.00	1.00
J	Estimated value ; the analyte concentration was less than the LOQ.				

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Lab Report #: L13080428
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-11	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/13/2013 20:36
Collect Date: 08/07/2013 14:20	Dilution: 2	File ID: TC08122013.066
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		3.74	J	2.00	1.00

Sample #: L13080428-11	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: 07/09/2013 14:51
Workgroup #: WG440968	Analyst: DIH	Run Date: 08/13/2013 20:36
Collect Date: 08/07/2013 14:20	Dilution: 2	File ID: TC08122013.066
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.27	J	2.00	1.00

Sample #: L13080428-12	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:02
Collect Date: 08/07/2013 14:20	Dilution: 2	File ID: TC08122013.044
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		48.1	J	2.00	1.00

Sample #: L13080428-12	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: 07/09/2013 14:51
Workgroup #: WG440969	Analyst: DIH	Run Date: 08/13/2013 16:02
Collect Date: 08/07/2013 14:20	Dilution: 2	File ID: TC08122013.044
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	5.71	B	2.00	1.00



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13080428

DATE: January 10, 2014

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, 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
54MW09	L13080428-01	54MW10	L13080428-07
54MW11	L13080428-03	54RB080713	L13080428-09
54MW08	L13080428-05	54MW12	L13080428-11

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.

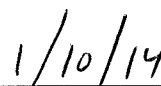
Table 1 Laboratory Performance Criteria

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



Date

**RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13080428**

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 08/07/13, the coolers were received on 08/08/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C and 3.0°C. All criteria were met. No qualifiers were applied.
- Holding Time Review: The aqueous samples were collected on 08/07/13 for perchlorate analysis. The aqueous samples were prepped and analyzed on 08/13/13. 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, ¹⁸O-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 $RSD \leq 20\%$ DoD Perchlorate Handbook)
 ICV ($\leq 15\%$ DoD Perchlorate Handbook)
 CCV/ICS ($\leq 15\%$ DoD Perchlorate Handbook)
 LODV ($\pm 30\%$ DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 07/12/13 on instrument LCMS1, all criteria were met for all target compounds ($RSD \leq 20\%$). 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 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), and 54MW12 (L13080428-11) apply to this initial and continuing calibrations.

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 54RB080713 (L13080428-09) 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)
08/13/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
08/13/13	WG441246-02	All perchlorate <½MRL	NA	NA	None
08/13/13	54RB080713	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 WG441246-03 was used as aqueous LCS for perchlorate analysis dated 08/13/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), and 54MW12 (L13080428-11) 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%.

- Sample 54MW08 (L13080428-05) was used as aqueous MS/MSD for perchlorate analysis dated 08/13/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), and 54MW12 (L13080428-11) 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.

- 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: 54MW09 (L13080428-01), 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:

$$m = 1.37$$

$$b = 0.00457$$

$$Y = \text{Area} = 25900/276000 = 0.09384$$

$$X = 0.06516$$

$$\text{Conc. } \mu\text{g/L} = (\text{Ax} * \text{Cis} * \text{DF})$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard

Cis = Conc. Of internal Standard ($\mu\text{g/L}$)

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (0.06156 * 5 * 1) = 0.326 \mu\text{g/L (Signal \#1)}$$

Reported Value = 0.327 $\mu\text{g/L}$

% Difference = 0.3%

Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

²The 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 #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-01	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW09	Prep Method: 6850	Prep Date: 08/13/2013 16:15
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441246	Analyst: JWR	Run Date: 08/13/2013 19:05
Collect Date: 08/07/2013 09:50	Dilution: 1	File ID: 1LM.LM22072
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.327		0.200	0.100

Sample #: L13080428-03	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW11	Prep Method: 6850	Prep Date: 08/13/2013 16:15
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441246	Analyst: JWR	Run Date: 08/13/2013 19:24
Collect Date: 08/07/2013 10:55	Dilution: 1	File ID: 1LM.LM22073
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.132	J J	0.200	0.100
J	Estimated value ; the analyte concentration was less than the LOQ.				

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Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-05	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW08	Prep Method: 6850	Prep Date: 08/13/2013 16:15
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441246	Analyst: JWR	Run Date: 08/13/2013 18:08
Collect Date: 08/07/2013 12:05	Dilution: 1	File ID: 1LM.LM22069
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.334		0.200	0.100

Sample #: L13080428-07	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW10	Prep Method: 6850	Prep Date: 08/13/2013 16:15
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441246	Analyst: JWR	Run Date: 08/13/2013 19:42
Collect Date: 08/07/2013 13:15	Dilution: 1	File ID: 1LM.LM22074
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.365		0.200	0.100

Sample #: L13080428-09	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54RB080713	Prep Method: 6850	Prep Date: 08/13/2013 16:15
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441246	Analyst: JWR	Run Date: 08/13/2013 21:36
Collect Date: 08/07/2013 13:35	Dilution: 1	File ID: 1LM.LM22080
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 #: L13080428

Lab Project #: 2773.087

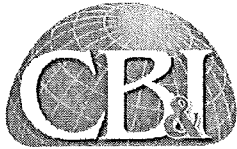
Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-11	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW12	Prep Method: 6850	Prep Date: 08/13/2013 16:15
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG441246	Analyst: JWR	Run Date: 08/13/2013 20:20
Collect Date: 08/07/2013 14:20	Dilution: 10	File ID: 1LM.LM22076
Sample Tag: DL01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	9.88		2.00	1.00



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13080428

DATE: January 10, 2014

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, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of seven aqueous samples (includes one dilution sample) were validated. The sample IDs are:


Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW09	L13080428-01	54RB080713	L13080428-09
54MW11	L13080428-03	54MW12	L13080428-11
54MW08	L13080428-05	54MW12	L13080428-11DL
54MW10	L13080428-07		

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, except for the following. For sample 54MW12 (L13080428-11), target compound 2,4,6-trinitrotoluene exceeded calibration range. Sample 54MW12 (L13080428-11DL) should be used for this compound. There were no impacts for data usability based on this outlier.



 Eric Malarek, Chemist

1/10/14

 Date

**RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13080428**

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 08/07/13, the coolers were received on 08/08/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C and 3.0°C. All criteria were met. No qualifiers were applied.
- Holding Time Review: The aqueous samples were collected on 08/07/13. The samples were extracted on 08/14/13 and were analyzed on 08/15/13 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 54RB080713 (L13080428-09) 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)
08/15/13	WG441278-01	All target explosives <½MRL	NA	NA	None
08/15/13	54RB080713	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 $\leq 20\%$.

- 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 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), 54MW12 (L13080428-11), and 54MW12 (L13080428-11DL) 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 $\pm 20\%$.

- 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 field samples were reported using this initial verification calibration.
- For explosives continuing calibration performed on 08/14/13 @23:07 on instrument HPLC5, all criteria were met. No qualifiers were applied. Field samples 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), and 54MW12 (L13080428-11) were reported using this continuing calibration.
- For explosives continuing calibration performed on 08/15/13 @07:14 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous field samples were reported using this continuing calibration.
- For explosives continuing calibration performed on 08/15/13 @11:36 on instrument HPLC5, all criteria were met. No qualifiers were applied. Sample 54MW12 (L13080428-11DL) was reported using this continuing calibration.
- For explosives continuing calibration performed on 08/15/13 @19:17 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous field 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

Laboratory control samples (LCS) 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.

- Sample WG441278-02 was used as the aqueous LCS for the explosives analysis on 08/15/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), 54MW12 (L13080428-11), and 54MW12 (L13080428-11DL) 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 $\leq 20\%$ RPD.

- No site specific aqueous MS/MSD was performed for the explosives analysis on 08/15/13; therefore, were not evaluated.

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 54MW12 (L13080428-11), target compound 2,4,6-trinitrotoluene exceeded calibration range. Sample 54MW12 (L13080428-11DL) should be used for this compound. There were no impacts for data usability based on this outlier.
- All positive detections were confirmed by mass spectrum (MS) analysis. All MS confirmations were within criteria for all detected target analytes in this SDG. No qualifiers were applied.

Sample: 54MW12 (L13080428-11), 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:

$$m = 0.28909729$$

$$b = -1.8106455$$

$$Y = \text{Area} = 122.26541$$

$$X = 429.18$$

$$\text{Conc. } \mu\text{g/L} = (A_x * V_t * DF) / (V_s)$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

A_x = Amount of compound being measured ($\mu\text{g/L}$).

V_t = Volume of total extract (mL) from bench sheet.

V_s = Volume of sample extracted (mL).

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (429.18 * 10 * 1) / (860) = 4.99 \mu\text{g/L (Signal \#1)}$$

Reported Value = 4.99 $\mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

²The 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 #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-01

PrePrep Method: N/A

Instrument: HPLC5

Client ID: 54MW09

Prep Method: METHOD

Prep Date: 08/14/2013 08:00

Matrix: Water

Analytical Method: 8330B

Cal Date: 03/20/2013 17:01

Workgroup #: WG441348

Analyst: JWR

Run Date: 08/15/2013 01:04

Collect Date: 08/07/2013 09:50

Dilution: 1

File ID: 5L010796.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	91.3	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

Microbac

Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-03

Client ID: 54MW11

Matrix: Water

Workgroup #: WG441348

Collect Date: 08/07/2013 10:55

Sample Tag: 01

PrePrep Method: N/A

Prep Method: METHOD

Analytical Method: 8330B

Analyst: JWR

Dilution: 1

Units: ug/L

Instrument: HPLC5

Prep Date: 08/14/2013 08:00

Cal Date: 03/20/2013 17:01

Run Date: 08/15/2013 01:43

File ID: 5L010797.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	94.3	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

Microbac

Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-05	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54MW08	Prep Method: METHOD	Prep Date: 08/14/2013 08:00
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441348	Analyst: JWR	Run Date: 08/15/2013 02:22
Collect Date: 08/07/2013 12:05	Dilution: 1	File ID: 5L010798.F
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.25	0.313
2,4-Dinitrotoluene	121-14-2		U	1.25	0.313
2,6-Dinitrotoluene	606-20-2		U	1.25	0.313
2,4,6-Trinitrotoluene	118-96-7		U	1.25	0.313
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.25	0.313
2-Nitrotoluene	88-72-2		U	1.25	0.313
4-Nitrotoluene	99-99-0		U	1.25	0.313
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.25	0.313
RDX	121-82-4		U	1.25	0.313
Nitroglycerin	55-63-0		U	1.25	0.313
Surrogate	Recovery	Lower Limit	Upper Limit	Q	
1,2-Dinitrobenzene	91.6	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

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Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-07	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54MW10	Prep Method: METHOD	Prep Date: 08/14/2013 08:00
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441348	Analyst: JWR	Run Date: 08/15/2013 03:01
Collect Date: 08/07/2013 13:15	Dilution: 1	File ID: 5L010799.F
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.25	0.313
2,4-Dinitrotoluene	121-14-2		U	1.25	0.313
2,6-Dinitrotoluene	606-20-2		U	1.25	0.313
2,4,6-Trinitrotoluene	118-96-7		U	1.25	0.313
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.25	0.313
2-Nitrotoluene	88-72-2		U	1.25	0.313
4-Nitrotoluene	99-99-0		U	1.25	0.313
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.25	0.313
RDX	121-82-4	2.23		1.25	0.313
Nitroglycerin	55-63-0		U	1.25	0.313
Surrogate	Recovery	Lower Limit	Upper Limit	Q	
1,2-Dinitrobenzene	88.2	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

Microbac

Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-09
 Client ID: 54RB080713
 Matrix: Water
 Workgroup #: WG441348
 Collect Date: 08/07/2013 13:35
 Sample Tag: 01

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

Instrument: HPLC5
 Prep Date: 08/14/2013 08:00
 Cal Date: 03/20/2013 17:01
 Run Date: 08/15/2013 03:41
 File ID: 5L010800.F

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.20	0.301
2,4-Dinitrotoluene	121-14-2		U	1.20	0.301
2,6-Dinitrotoluene	606-20-2		U	1.20	0.301
2,4,6-Trinitrotoluene	118-96-7		U	1.20	0.301
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.20	0.301
2-Nitrotoluene	88-72-2		U	1.20	0.301
4-Nitrotoluene	99-99-0		U	1.20	0.301
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.20	0.301
RDX	121-82-4		U	1.20	0.301
Nitroglycerin	55-63-0		U	1.20	0.301
Surrogate	Recovery	Lower Limit	Upper Limit	Q	
1,2-Dinitrobenzene	97.7	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

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Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-11	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54MW12	Prep Method: METHOD	Prep Date: 08/14/2013 08:00
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441348	Analyst: JWR	Run Date: 08/15/2013 04:20
Collect Date: 08/07/2013 14:20	Dilution: 1	File ID: 5L010801.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	65.9	J R	1.16	0.291
2-Amino-4,6-dinitrotoluene	35572-78-2	8.67		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	4.99		1.16	0.291
RDX	121-82-4	14.6		1.16	0.291
Nitroglycerin	55-63-0		U	1.16	0.291

Surrogate	Recovery	Lower Limit	Upper Limit	Q
1,2-Dinitrobenzene	98.0	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.			

Microbac

Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-11	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54MW12	Prep Method: METHOD	Prep Date: 08/14/2013 08:00
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441348	Analyst: JWR	Run Date: 08/15/2013 13:07
Collect Date: 08/07/2013 14:20	Dilution: 5	File ID: 5L010810.F
Sample Tag: DL01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	5.81	1.45
2,4-Dinitrotoluene	121-14-2		U	5.81	1.45
2,6-Dinitrotoluene	606-20-2		U	5.81	1.45
2,4,6-Trinitrotoluene	118-96-7	65.9		5.81	1.45
2-Amino-4,6-dinitrotoluene	35572-78-2	8.65	J J	5.81	1.45
2-Nitrotoluene	88-72-2			5.81	1.45
4-Nitrotoluene	99-99-0			5.81	1.45
4-Amino-2,6-dinitrotoluene	19406-51-0	5.18	J J	5.81	1.45
RDX	121-82-4	14.5	J J	5.81	1.45
Nitroglycerin	55-63-0			5.81	1.45
Surrogate	Recovery	Lower Limit	Upper Limit	Q	
1,2-Dinitrobenzene	79.3	50	150		
J	The reported result is an estimated value.				
U	Analyte was not detected. The concentration is below the reported LOD.				



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13080428

DATE: January 24, 2014

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, 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
54MW09	L13080428-01	54MW10	L13080428-07
54MW11	L13080428-03	54RB080713	L13080428-09
54MW08	L13080428-05	54MW12	L13080428-11

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

1/24/14

 Date

**RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13080428**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 28 days for sulfate and chloride and Cool to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ with H_2SO_4 to $\text{pH} < 2$ 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 08/07/13, the coolers were received on 08/08/13 by the primary laboratory (Microbac Laboratories, Inc.) at 4.0°C and 3.0°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: Samples were collected on 08/07/13. The samples were prepped and analyzed on 08/09/13 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 07/23/13 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 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), and 54MW12 (L13080428-11) 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 54RB080713 (L13080428-09) 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)
08/09/13	Chloride	ICB/CCBs	<LOD	NA	None
08/09/13	Sulfate	ICB/CCBs	<LOD	NA	None
08/09/13	Nitrate	ICB/CCBs	<LOD	NA	None
08/09/13	Chloride	WG440725-01	<½MRL	NA	None
08/09/13	Sulfate	WG440725-01	<½MRL	NA	None
08/09/13	Nitrate	WG440725-01	<½MRL	NA	None
08/09/13	Chloride	54RB080713	0.117	0.585	None
08/09/13	Sulfate	54RB080713	<½MRL	NA	None
08/09/13	Nitrate	54RB080713	<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 WG440725-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 08/09/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), and 54MW12 (L13080428-11) 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%)

- Sample 54RB080713 (L13080428-09) was used as the aqueous MS/MSD for chloride, sulfate, and nitrate analysis on 08/09/13. All criteria were met. No qualifiers were applied. Samples 54MW09 (L13080428-01), 54MW11 (L13080428-03), 54MW08 (L13080428-05), 54MW10 (L13080428-07), 54RB080713 (L13080428-09), and 54MW12 (L13080428-11) 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 analysis 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: 54MW09 (L13080428-01), sulfate

$$Y = mX + b$$

Y = Sample Area

m = slope of curve

X = Concentration (mg/L)

b = Y-intercept

DF = Dilution Factor

Given:

$$m = 0.1355$$

$$b = -0.0194$$

$$Y = \text{Area} = 5.286$$

$$DF = 1$$

$$X = 39.2 \text{ mg/L} * DF = 39.2 \text{ mg/L} * 1 = 39.2 \text{ mg/L}$$

$$\text{Reported concentration} = 39.2 \text{ mg/L}$$

$$\%D = 0.0\%$$

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Microbac

Lab Report #: L13080428
Lab Project #: 2773.087
Project Name: Radford
Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-01	PrePrep Method: N/A	Instrument: IC2
Client ID: 54MW09	Prep Method: 9056	Prep Date: 08/08/2013 15:00
Matrix: Water	Analytical Method: 9056	Cal Date: 07/23/2013 18:03
Workgroup #: WG440725	Analyst: KRB	Run Date: 08/09/2013 00:29
Collect Date: 08/07/2013 09:50	Dilution: 1	File ID: I2_080813-51
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.32		0.200	0.100
Nitrate	14797-55-8	0.194	J	0.600	0.100
Sulfate	14808-79-8	39.2		1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

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Lab Report #: L13080428
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-03	PrePrep Method: N/A	Instrument: IC2
Client ID: 54MW11	Prep Method: 9056	Prep Date: 08/08/2013 15:00
Matrix: Water	Analytical Method: 9056	Cal Date: 07/23/2013 18:03
Workgroup #: WG440725	Analyst: KRB	Run Date: 08/09/2013 00:48
Collect Date: 08/07/2013 10:55	Dilution: 1	File ID: I2_080813-52
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.13		0.200	0.100
Nitrate	14797-55-8	0.120	J J	0.600	0.100
Sulfate	14808-79-8	91.8		1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Microbac

Lab Report #: L13080428
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-05	PrePrep Method: N/A	Instrument: IC2
Client ID: 54MW08	Prep Method: 9056	Prep Date: 08/08/2013 15:00
Matrix: Water	Analytical Method: 9056	Cal Date: 07/23/2013 18:03
Workgroup #: WG440725	Analyst: KRB	Run Date: 08/09/2013 01:26
Collect Date: 08/07/2013 12:05	Dilution: 1	File ID: I2_080813-54
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.54		0.200	0.100
Nitrate	14797-55-8	0.273	J J	0.600	0.100
Sulfate	14808-79-8	38.0		1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Microbac

Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #:	L13080428-07	PrePrep Method:	N/A	Instrument:	IC2
Client ID:	54MW10	Prep Method:	9056	Prep Date:	08/08/2013 15:00
Matrix:	Water	Analytical Method:	9056	Cal Date:	07/23/2013 18:03
Workgroup #:	WG440725	Analyst:	KRB	Run Date:	08/09/2013 01:44
Collect Date:	08/07/2013 13:15	Dilution:	1	File ID:	I2_080813-55
Sample Tag:	01	Units:	mg/L		

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.83		0.200	0.100
Nitrate	14797-55-8	0.206	J J	0.600	0.100
Sulfate	14808-79-8	44.3		1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Form I Copy

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Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-09

PrePrep Method: N/A

Instrument: IC2

Client ID: 54RB080713

Prep Method: 9056

Prep Date: 08/08/2013 15:00

Matrix: Water

Analytical Method: 9056

Cal Date: 07/23/2013 18:03

Workgroup #: WG440725

Analyst: KRB

Run Date: 08/09/2013 02:03

Collect Date: 08/07/2013 13:35

Dilution: 1

File ID: I2_080813-56

Sample Tag: 01

Units: mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	0.117	J J	0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8		U	1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				
U	Analyte was not detected. The concentration is below the reported LOD.				

Form I Copy

Microbac

Lab Report #: L13080428

Lab Project #: 2773.087

Project Name: Radford

Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080428-11	PrePrep Method: N/A	Instrument: IC2
Client ID: 54MW12	Prep Method: 9056	Prep Date: 08/08/2013 15:00
Matrix: Water	Analytical Method: 9056	Cal Date: 07/23/2013 18:03
Workgroup #: WG440725	Analyst: KRB	Run Date: 08/09/2013 03:56
Collect Date: 08/07/2013 14:20	Dilution: 1	File ID: I2_080813-62
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	5.03		0.200	0.100
Nitrate	14797-55-8	4.80		0.600	0.100
Sulfate	14808-79-8	39.6		1.00	0.500



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon
Microbac Laboratories, Inc. L13080274

DATE: December 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 August 5, 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
54MW13	L13080274-01	54MW04	L13080274-07
54MW13	L13080274-02	54MW04	L13080274-08
54MW01	L13080274-03	54MW14	L13080274-09
54MW01	L13080274-04	54MW14	L13080274-10
54TM01	L13080274-05	54TM14	L13080274-11
54TM01	L13080274-06	54TM14	L13080274-12

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

12/19/13

 Date

**RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG L13080274**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, 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 08/05/13, the coolers were received on 08/06/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 1.0°C . Even though one of the receipt temperatures was below criteria for the coolers; 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 08/05/13. The TOC and TIC analysis were run on 08/07/13. The DOC and DIC analysis were run on 08/09/13. 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 08/07/13. The DOC and DIC analysis were run on 08/09/13. The initial calibration for TC was analyzed on 07/09/13 with a coefficient of determination of 0.9996. The initial calibration for TIC was analyzed on 07/09/13 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 (L13080274-01), 54MW13 (L13080274-02), 54MW01 (L13080274-03), 54MW01 (L13080274-04), 54TM01 (L13080274-05), 54TM01 (L13080274-06), 54MW04 (L13080274-07), 54MW04 (L13080274-08), 54MW14 (L13080274-09), 54MW14 (L13080274-10), 54TM14 (L13080274-11), and 54TM14 (L13080274-12) 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 ½MRL (<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 54RB080713 (L13080428-09) applies to all of the dissolved samples (DOC and DIC) in this SDG and rinse blank 54RB080713 (L13080428-09) 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)
08/07/13	TOC	ICB/CCBs	<LOD	NA	None
08/07/13	TOC	ICB/CCBs	<LOD	NA	None
08/07/13	TOC	WG440410-01	<½MRL	NA	None
08/08/13	DOC	WG440411-01	<½MRL	NA	None
08/12/13	TOC	54RB080713	0.607J	3.04	54MW13, 54TM01, 54MW04
08/12/13	TIC	54RB080713	<½MRL	NA	None
08/13/13	DOC	54RB080713	1.45J	7.25	54MW13, 54MW01, 54TM01, 54MW04, 54MW14, 54TM14
08/13/13	DIC	54RB080713	<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 WG440410-02 and WG440410-03 were used as the aqueous LCS/LCSD for TOC and TIC analysis on 08/07/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13080274-01), 54MW01 (L13080274-03), 54TM01 (L13080274-05), 54MW04 (L13080274-07), 54MW14 (L13080274-09), and 54TM14 (L13080274-11) apply to this LCS/LCSD.
- Samples WG440411-02 and WG440411-03 were used as the aqueous LCS/LCSD for DOC and DIC analysis on 08/08/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13080274-02), 54MW01 (L13080274-04), 54TM01 (L13080274-06), 54MW04 (L13080274-08), 54MW14 (L13080274-10), and 54TM14 (L13080274-12) 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 \leq 15%).

- No site specific aqueous MS/MSD was performed for TOC and TIC analysis on 08/07/13; 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 08/08/13; 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 (\leq 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.

- Field groundwater sample duplicate pair 54MW01 (L13080274-03; L13080274-04) and 54TM01 (L13080274-05; L13080274-06) was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG. TOC was detected at 3.32 mg/L in the parent sample and at 2.38 mg/L in the duplicate pair; resulting in a RPD of 33.0%. TIC was detected at 20.4 mg/L in the parent sample and at 16.8 mg/L in the duplicate pair; resulting in a RPD of 19.4%. DOC was detected at 3.74 mg/L in the parent sample and at 4.07 mg/L in the duplicate pair; resulting in a RPD of 8.5%. DIC was detected at 39.3 mg/L in the parent sample and at 37.7 mg/L in the duplicate pair; resulting in a RPD of 4.2%. TOC was qualified estimated "J" for the duplicate pair based upon the high RPD. All criteria were met for TIC, DOC, and DIC analysis.
- Field groundwater sample duplicate pair 54MW14 (L13080274-09; L13080274-10) and 54TM14 (L13080274-11; L13080274-12) was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG. TOC was detected at 3.64 mg/L in the parent sample and at 4.00 mg/L in the duplicate pair; resulting in a RPD of 9.4%. TIC was detected at 29.6 mg/L in the parent sample and at 30.3 mg/L in the duplicate pair; resulting in a RPD of 2.3%. DOC was detected at 4.36 mg/L in the parent sample and at 4.13 mg/L in the duplicate pair; resulting in a RPD of 5.4%. DIC was detected at 51.3 mg/L in the parent sample and at 45.4 mg/L in the duplicate pair; resulting in a RPD of 12.2%. All criteria were met for TOC, TIC, DOC, and DIC analysis. 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 54MW13 (L13080274-01), 54MW13 (L13080274-02), 54MW01 (L13080274-03), 54MW01 (L13080274-04), 54TM01 (L13080274-05), 54TM01 (L13080274-06), 54MW04 (L13080274-07), 54MW04 (L13080274-08), 54MW14 (L13080274-09), 54MW14 (L13080274-10), 54TM14 (L13080274-11), and 54TM14 (L13080274-12) 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	1.0; 2.0	2.35	4.02	41.5	J
54MW13	TIC/DIC	1.0; 2.0	24.5	38.1	35.7	J
54MW01	TOC/DOC	1.0; 2.0	3.32	3.74	11.2	J
54MW01	TIC/DIC	1.0; 2.0	20.4	39.3	48.1	J
54TM01	TOC/DOC	1.0; 2.0	2.38	4.07	41.5	J
54TM01	TIC/DIC	1.0; 2.0	16.8	37.7	55.4	J
54MW04	TOC/DOC	1.0; 2.0	2.70	3.19	15.4	J
54MW04	TIC/DIC	1.0; 2.0	20.6	44.8	54.0	J
54MW14	TOC/DOC	1.0; 2.0	3.64	4.36	16.5	J
54MW14	TIC/DIC	1.0; 2.0	29.6	51.3	42.3	J
54TM14	TOC/DOC	1.0; 2.0	4.00	4.13	3.2	None
54TM14	TIC/DIC	1.0; 2.0	30.3	45.4	33.3	J

J = Estimated value.

MRL = Method Reporting Limit

NA = Not Applicable

Sample: 54MW13 (L13080274-01), TOC

$$TC: Y = m \cdot X \text{ (mg/L)} + b$$

$$TIC: Y = m \cdot X \text{ (mg/L)} + b$$

$$m = 36.20$$

$$m = 29.78$$

$$b = 6.332$$

$$b = 8.713$$

$$Y = 978.8$$

$$Y = 738.8$$

$$DF = 1$$

$$DF = 1$$

$$X = (26.86 \text{ mg/L}) \cdot 1 = 26.86 \text{ mg/L}$$

$$X = (24.52 \text{ mg/L}) \cdot 1 = 24.52 \text{ mg/L}$$

$$TOC \text{ (mg/L)} = TC \text{ (mg/L)} - TIC \text{ (mg/L)} = 26.86 - 24.52 = 2.34 \text{ mg/L}$$

Reported Value = 2.35 mg/L

% Difference = 0.4%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Certificate of Analysis

Sample #: L13080274-01	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 16:01
Collect Date: 08/05/2013 09:10	Dilution: 1	File ID: TC08072013.012
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.35	B	1.00	0.500

Sample #: L13080274-01	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 16:01
Collect Date: 08/05/2013 09:10	Dilution: 1	File ID: TC08072013.012
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		24.5	J	1.00	0.500

Sample #: L13080274-02	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:02
Collect Date: 08/05/2013 09:10	Dilution: 2	File ID: TC08082013.049
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		38.1	J	2.00	1.00

Sample #: L13080274-02	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:02
Collect Date: 08/05/2013 09:10	Dilution: 2	File ID: TC08082013.049
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.02	B	2.00	1.00

Certificate of Analysis

Sample #: L13080274-03	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 16:15
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: TC08072013.013
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.32	J	1.00	0.500

Sample #: L13080274-03	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 16:15
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: TC08072013.013
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		20.4	J	1.00	0.500

Sample #: L13080274-04	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:16
Collect Date: 08/05/2013 10:40	Dilution: 2	File ID: TC08082013.050
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.74	J	2.00	1.00

Sample #: L13080274-04	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:16
Collect Date: 08/05/2013 10:40	Dilution: 2	File ID: TC08082013.050
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		39.3	B	2.00	1.00

Certificate of Analysis

Sample #: L13080274-05	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 16:49
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: TC08072013.016
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.38	B	1.00	0.500

Sample #: L13080274-05	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 16:49
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: TC08072013.016
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		16.8	J	1.00	0.500

Sample #: L13080274-06	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:29
Collect Date: 08/05/2013 10:40	Dilution: 2	File ID: TC08082013.051
Sample Tag: DL01	Units: mg/L	

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

Sample #: L13080274-06	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:29
Collect Date: 08/05/2013 10:40	Dilution: 2	File ID: TC08082013.051
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.07	B	2.00	1.00

Certificate of Analysis

Sample #: L13080274-07	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 17:02
Collect Date: 08/05/2013 12:15	Dilution: 1	File ID: TC08072013.017
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	2.70	B	1.00	0.500

Sample #: L13080274-07	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 17:02
Collect Date: 08/05/2013 12:15	Dilution: 1	File ID: TC08072013.017
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		20.6	J	1.00	0.500

Sample #: L13080274-08	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:42
Collect Date: 08/05/2013 12:15	Dilution: 2	File ID: TC08082013.052
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		44.8	J	2.00	1.00

Sample #: L13080274-08	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:42
Collect Date: 08/05/2013 12:15	Dilution: 2	File ID: TC08082013.052
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	3.19	B	2.00	1.00

Certificate of Analysis

Sample #: L13080274-09	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 17:15
Collect Date: 08/05/2013 13:35	Dilution: 1	File ID: TC08072013.018
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		29.6	J	1.00	0.500

Sample #: L13080274-09	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: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 17:15
Collect Date: 08/05/2013 13:35	Dilution: 1	File ID: TC08072013.018
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	3.64	J	1.00	0.500

Sample #: L13080274-10	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:55
Collect Date: 08/05/2013 13:35	Dilution: 2	File ID: TC08082013.053
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		51.3	J	2.00	1.00

Sample #: L13080274-10	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: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 13:55
Collect Date: 08/05/2013 13:35	Dilution: 2	File ID: TC08082013.053
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.36	B	2.00	1.00

Certificate of Analysis

Sample #: L13080274-11	PrePrep Method: N/A	Instrument: TOC-VWP
Client ID: 54TM14	Prep Method: 415.1	Prep Date: N/A
Matrix: Water	Analytical Method: 415.1	Cal Date: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 17:29
Collect Date: 08/05/2013 13:35	Dilution: 1	File ID: TC08072013.019
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Organic Carbon	TOC	4.00		1.00	0.500

Sample #: L13080274-11	PrePrep Method: N/A	Instrument: TOC-VWP
Client ID: 54TM14	Prep Method: 415.1	Prep Date: N/A
Matrix: Water	Analytical Method: 415.1	Cal Date: 07/09/2013 14:51
Workgroup #: WG440410	Analyst: DIH	Run Date: 08/07/2013 17:29
Collect Date: 08/05/2013 13:35	Dilution: 1	File ID: TC08072013.019
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Total Inorganic Carbon		30.3	J	1.00	0.500

Sample #: L13080274-12	PrePrep Method: N/A	Instrument: TOC-VWP
Client ID: 54TM14	Prep Method: 415.1	Prep Date: N/A
Matrix: Water	Analytical Method: 415.1	Cal Date: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 14:09
Collect Date: 08/05/2013 13:35	Dilution: 2	File ID: TC08082013.054
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Organic Carbon, Dissolved	TOC	4.13	B	2.00	1.00

Sample #: L13080274-12	PrePrep Method: N/A	Instrument: TOC-VWP
Client ID: 54TM14	Prep Method: 415.1	Prep Date: N/A
Matrix: Water	Analytical Method: 415.1	Cal Date: 07/09/2013 14:51
Workgroup #: WG440411	Analyst: DIH	Run Date: 08/09/2013 14:09
Collect Date: 08/05/2013 13:35	Dilution: 2	File ID: TC08082013.054
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Inorganic Carbon, Dissolved		45.4	J	2.00	1.00



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Microbac Laboratories, Inc. L13080274

DATE: December 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 August 5, 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
54MW13	L13080274-01	54MW04	L13080274-07
54MW01	L13080274-03	54MW14	L13080274-09
54TM01	L13080274-05	54TM14	L13080274-11

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.

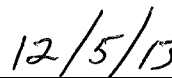
Table 1 Laboratory Performance Criteria

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



Date

**RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG L13080274**

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 08/05/13, the coolers were received on 08/06/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 1.0°C. Even though one of the receipt temperatures was below criteria for the coolers; 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 08/05/13 for perchlorate analysis. The aqueous samples were prepped and analyzed on 08/09/13. 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, ¹⁸O-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 07/12/13 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 (L13080274-01), 54MW01 (L13080274-03), 54TM01 (L13080274-05), 54MW04 (L13080274-07), 54MW14 (L13080274-09), and 54TM14 (L13080274-11) apply to this initial and continuing calibrations.

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 54RB080713 (L13080428-09) 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)
08/09/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
08/09/13	WG440826-02	All perchlorate <½MRL	NA	NA	None
08/13/13	54RB080713	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 WG440826-03 was used as aqueous LCS for perchlorate analysis dated 08/09/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13080274-01), 54MW01 (L13080274-03), 54TM01 (L13080274-05), 54MW04 (L13080274-07), 54MW14 (L13080274-09), and 54TM14 (L13080274-11) 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 performed for perchlorate analysis on 08/09/13; 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.

- Field groundwater sample duplicate pair 54MW01 (L13080274-03) and 54TM01 (L13080274-05) was analyzed for perchlorate analysis in this SDG. Perchlorate was non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.
- Field groundwater sample duplicate pair 54MW14 (L13080274-09) and 54TM14 (L13080274-11) was analyzed for perchlorate analysis in this SDG. Perchlorate was detected in the parent sample at 0.456 µg/L and 0.489 µg/L for the duplicate pair; resulting in a RPD of 7.0%. 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: 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:

$$m = 1.37$$

$$b = 0.00457$$

$$Y = \text{Area} = 47700/354000 = 0.1347$$

$$X = 0.09502$$

$$\text{Conc. } \mu\text{g/L} = (\text{Ax} * \text{Cis} * \text{DF})$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard

Cis = Conc. Of internal Standard ($\mu\text{g/L}$)

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (0.09502 * 5 * 1) = 0.475 \mu\text{g/L (Signal \#1)}$$

Reported Value = 0.477 $\mu\text{g/L}$

% Difference = 0.4%

Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Certificate of Analysis

Sample #: L13080274-01	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW13	Prep Method: 6850	Prep Date: 08/09/2013 15:00
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG440826	Analyst: JWR	Run Date: 08/09/2013 21:44
Collect Date: 08/05/2013 09:10	Dilution: 1	File ID: 1LM.LM22030
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.477		0.200	0.100

Sample #: L13080274-03	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54MW01	Prep Method: 6850	Prep Date: 08/09/2013 15:00
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG440826	Analyst: JWR	Run Date: 08/09/2013 22:03
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: 1LM.LM22031
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.				

Certificate of Analysis

Sample #: L13080274-05	PrePrep Method: N/A	Instrument: LCMS1
Client ID: 54TM01	Prep Method: 6850	Prep Date: 08/09/2013 15:00
Matrix: Water	Analytical Method: 6850	Cal Date: 07/12/2013 13:15
Workgroup #: WG440826	Analyst: JWR	Run Date: 08/09/2013 22:22
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: 1LM.LM22032
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.				

Certificate of Analysis

Sample #: L13080274-07

PrePrep Method: N/A

Instrument: LCMS1

Client ID: 54MW04

Prep Method: 6850

Prep Date: 08/09/2013 15:00

Matrix: Water

Analytical Method: 6850

Cal Date: 07/12/2013 13:15

Workgroup #: WG440826

Analyst: JWR

Run Date: 08/09/2013 22:40

Collect Date: 08/05/2013 12:15

Dilution: 1

File ID: 1LM.LM22033

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.				

Certificate of Analysis

Sample #: L13080274-09 PrePrep Method: N/A Instrument: LCMS1
Client ID: 54MW14 Prep Method: 6850 Prep Date: 08/09/2013 15:00
Matrix: Water Analytical Method: 6850 Cal Date: 07/12/2013 13:15
Workgroup #: WG440826 Analyst: JWR Run Date: 08/09/2013 22:59
Collect Date: 08/05/2013 13:35 Dilution: 1 File ID: 1LM.LM22034
Sample Tag: 01 Units: ug/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.456		0.200	0.100

Sample #: L13080274-11 PrePrep Method: N/A Instrument: LCMS1
Client ID: 54TM14 Prep Method: 6850 Prep Date: 08/09/2013 15:00
Matrix: Water Analytical Method: 6850 Cal Date: 07/12/2013 13:15
Workgroup #: WG440826 Analyst: JWR Run Date: 08/09/2013 23:18
Collect Date: 08/05/2013 13:35 Dilution: 1 File ID: 1LM.LM22035
Sample Tag: 01 Units: ug/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Perchlorate	14797-73-0	0.489		0.200	0.100



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Microbac Laboratories, Inc, SDG L13080274

DATE: December 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 August 5, 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
54MW13	L13080274-01	54MW04	L13080274-07
54MW01	L13080274-03	54MW14	L13080274-09
54TM01	L13080274-05	54TM14	L13080274-11

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 with noted qualifications.



Eric Malarek, Chemist

12/5/13

Date

**RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG L13080274**

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 ($@4^{\circ}\text{C} \pm 2^{\circ}\text{C}$) 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 08/05/13, the coolers were received on 08/06/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 1.0°C . Even though one of the receipt temperatures was below criteria for the coolers; 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 08/05/13. The samples were extracted on 08/06/13 and were analyzed on 08/15/13 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 54RB080713 (L13080428-09) applies to all samples in this SDG.

Table 2 Blank Contamination Analysis Summary

Analysis Date	QC Blank ID	Compound	Max Conc. $\mu\text{g/L}$	Action Level $\mu\text{g/L}$	B qualified samples (For this SDG)
08/14/13	WG440305-01	All target explosives $< \frac{1}{2}\text{MRL}$	NA	NA	None
08/15/13	54RB080713	All target explosives $< \frac{1}{2}\text{MRL}$	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 $\leq 20\%$.

- 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 54MW13 (L13080274-01), 54MW01 (L13080274-03), 54TM01 (L13080274-05), 54MW04 (L13080274-07), 54MW14 (L13080274-09), and 54TM14 (L13080274-11) 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 $\pm 20\%$.

- 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 field samples were reported using this initial verification calibration.
- For explosives continuing calibration performed on 08/14/13 @15:01 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous field samples were reported using this continuing calibration.
- For explosives continuing calibration performed on 08/14/13 @23:07 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous field samples were reported using this continuing calibration.
- For explosives continuing calibration performed on 08/15/13 @11:36 on instrument HPLC5, all criteria were met. No qualifiers were applied. Samples 54MW13 (L13080274-01), 54MW01 (L13080274-03), 54TM01 (L13080274-05), 54MW04 (L13080274-07), 54MW14 (L13080274-09), and 54TM14 (L13080274-11) were reported using this continuing calibration.
- For explosives continuing calibration performed on 08/15/13 @19:17 on instrument HPLC5, all criteria were met. No qualifiers were applied. No aqueous field 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 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 and 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 $\leq 20\%$ RPD.

- Samples WG440305-02 and WG440305-03 were used as the aqueous LCS/LCSD for the explosives analysis on 08/14/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13080274-01), 54MW01 (L13080274-03), 54TM01 (L13080274-05), 54MW04 (L13080274-07), 54MW14 (L13080274-09), and 54TM14 (L13080274-11) 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 $\leq 20\%$ RPD.

- No site specific aqueous MS/MSD was performed for the explosives analysis on 08/15/13; therefore, were not evaluated.

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 (L13080274-03) and 54TM01 (L13080274-05) 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.
- Field groundwater sample duplicate pair 54MW14 (L13080274-09) and 54TM14 (L13080274-11) 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 positive detections were confirmed by mass spectrum (MS) analysis. All MS confirmations were within criteria for all detected target analytes in this SDG. No qualifiers were applied.

Sample: 54MW13 (L13080274-01), 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:

$$m = 0.28909729$$

$$b = -1.8106455$$

$$Y = \text{Area} = 15.73369$$

$$X = 60.69$$

$$\text{Conc. } \mu\text{g/L} = (A_x * V_t * DF) / (V_s)$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

A_x = Amount of compound being measured ($\mu\text{g/L}$).

V_t = Volume of total extract (mL) from bench sheet.

V_s = Volume of sample extracted (mL).

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (60.69 * 10 * 1) / (950) = 0.639 \mu\text{g/L (Signal \#1)}$$

Reported Value = 0.639 $\mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Certificate of Analysis

Sample #: L13080274-01

PrePrep Method: N/A

Instrument: HPLC5

Client ID: 54MW13

Prep Method: METHOD

Prep Date: 08/06/2013 09:01

Matrix: Water

Analytical Method: 8330B

Cal Date: 03/20/2013 17:01

Workgroup #: WG441341

Analyst: JWR

Run Date: 08/15/2013 13:46

Collect Date: 08/05/2013 09:10

Dilution: 1

File ID: 5L010811.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	3.81		1.05	0.263
2-Amino-4,6-dinitrotoluene	35572-78-2	0.798	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	0.639	J J	1.05	0.263
RDX	121-82-4	1.46		1.05	0.263
Nitroglycerin	55-63-0		U	1.05	0.263
Surrogate	Recovery	Lower Limit	Upper Limit	Q	
1,2-Dinitrobenzene	99.9	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.				

Certificate of Analysis

Sample #: L13080274-03

PrePrep Method: N/A

Instrument: HPLC5

Client ID: 54MW01

Prep Method: METHOD

Prep Date: 08/06/2013 09:01

Matrix: Water

Analytical Method: 8330B

Cal Date: 03/20/2013 17:01

Workgroup #: WG441341

Analyst: JWR

Run Date: 08/15/2013 14:25

Collect Date: 08/05/2013 10:40

Dilution: 1

File ID: 5L010812.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	93.9	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

Lab Report #: L13080274
 Lab Project #: 2773.087
 Project Name: Radford
 Lab Contact: Stephanie Mossburg

Certificate of Analysis

Sample #: L13080274-05	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54TM01	Prep Method: METHOD	Prep Date: 08/06/2013 09:01
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441341	Analyst: JWR	Run Date: 08/15/2013 15:04
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: 5L010813.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	94.0	50	150	
U	Analyte was not detected. The concentration is below the reported LOD.			

Certificate of Analysis

Sample #: L13080274-07	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54MW04	Prep Method: METHOD	Prep Date: 08/06/2013 09:01
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441341	Analyst: JWR	Run Date: 08/15/2013 15:43
Collect Date: 08/05/2013 12:15	Dilution: 1	File ID: 5L010814.F
Sample Tag: 01	Units: ug/L	

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	97.7	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				

Certificate of Analysis

Sample #: L13080274-09	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54MW14	Prep Method: METHOD	Prep Date: 08/06/2013 09:01
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441341	Analyst: JWR	Run Date: 08/15/2013 16:22
Collect Date: 08/05/2013 13:35	Dilution: 1	File ID: 5L010815.F
Sample Tag: 01	Units: ug/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
1,3-Dinitrobenzene	99-65-0		U	1.18	0.294
2,4-Dinitrotoluene	121-14-2		U	1.18	0.294
2,6-Dinitrotoluene	606-20-2		U	1.18	0.294
2,4,6-Trinitrotoluene	118-96-7		U	1.18	0.294
2-Amino-4,6-dinitrotoluene	35572-78-2		U	1.18	0.294
2-Nitrotoluene	88-72-2		U	1.18	0.294
4-Nitrotoluene	99-99-0		U	1.18	0.294
4-Amino-2,6-dinitrotoluene	19406-51-0		U	1.18	0.294
RDX	121-82-4		U	1.18	0.294
Nitroglycerin	55-63-0		U	1.18	0.294
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.				

Certificate of Analysis

Sample #: L13080274-11	PrePrep Method: N/A	Instrument: HPLC5
Client ID: 54TM14	Prep Method: METHOD	Prep Date: 08/06/2013 09:01
Matrix: Water	Analytical Method: 8330B	Cal Date: 03/20/2013 17:01
Workgroup #: WG441341	Analyst: JWR	Run Date: 08/15/2013 17:01
Collect Date: 08/05/2013 13:35	Dilution: 1	File ID: 5L010816.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	101	50	150		
U	Analyte was not detected. The concentration is below the reported LOD.				



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MEMORANDUM

TO: Tim Leahy, CFS Radford Army Ammunition Plant (RFAAP) Project Manager

FROM: Eric Malarek, CFS RFAAP Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Nitrate, and Sulfate
Microbac Laboratories, Inc. L13080274

DATE: December 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 August 5, 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
54MW13	L13080274-01	54MW04	L13080274-07DL
54MW01	L13080274-03	54MW14	L13080274-09
54TM01	L13080274-05	54TM14	L13080274-11
54MW04	L13080274-07		

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, except for the following. For sample 54MW04 (L13080274-07), sulfate exceeded calibration range. Sample 54MW04 (L13080274-07DL) should be used for this analyte. There were no impacts for data usability based on this outlier.


 Eric Malarek, Chemist

12/18/13
 Date

**RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG L13080274**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 28 days for sulfate and chloride and Cool to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ with H_2SO_4 to $\text{pH} < 2$ 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 08/05/13, the coolers were received on 08/06/13 by the primary laboratory (Microbac Laboratories, Inc.) at 3.0°C and 1.0°C . Even though one of the receipt temperatures was below criteria for the coolers; there were no impacts to the data quality and no qualifiers were applied based upon this outlier.
- Holding Time Review: Samples were collected on 08/05/13. The samples were prepped and analyzed on 08/06/13 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 05/01/13 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 54MW13 (L13080274-01), 54MW01 (L13080274-03), 54TM01 (L13080274-05), 54MW04 (L13080274-07), 54MW04 (L13080274-07DL), 54MW14 (L13080274-09), and 54TM14 (L13080274-11) 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 54RB080713 (L13080428-09) 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)
08/06/13	Chloride	ICB/CCBs	<LOD	NA	None
08/06/13	Sulfate	ICB/CCBs	<LOD	NA	None
08/06/13	Nitrate	ICB/CCBs	<LOD	NA	None
08/06/13	Chloride	WG440384-01	<½MRL	NA	None
08/06/13	Sulfate	WG440384-01	<½MRL	NA	None
08/06/13	Nitrate	WG440384-01	<½MRL	NA	None
08/09/13	Chloride	54RB080713	0.117	0.585	None
08/09/13	Sulfate	54RB080713	<½MRL	NA	None
08/09/13	Nitrate	54RB080713	<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 WG440384-02 was used as the aqueous LCS for chloride, sulfate, and nitrate analysis on 08/06/13. All criteria were met. No qualifiers were applied. Samples 54MW13 (L13080274-01), 54MW01 (L13080274-03), 54TM01 (L13080274-05), 54MW04 (L13080274-07), 54MW04 (L13080274-07DL), 54MW14 (L13080274-09), and 54TM14 (L13080274-11) 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 sample was used as the aqueous MS/MSD for chloride, sulfate, and nitrate analysis on 08/06/13; therefore, was not evaluated.

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 54MW01 (L13080274-03) and 54TM01 (L13080274-05) was analyzed for chloride, sulfate, and nitrate analysis in this SDG. Chloride was detected at 1.64 mg/L in the parent sample and at 1.67 mg/L in the duplicate pair; resulting in a RPD of 1.8%. Sulfate was detected at 26.7 mg/L in the parent sample and at 27.0 mg/L in the duplicate pair; resulting in a RPD of 1.1%. Nitrate was non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.
- Field groundwater sample duplicate pair 54MW14 (L13080274-09) and 54TM14 (L13080274-11) was analyzed for chloride, sulfate, and nitrate analysis in this SDG. Chloride was detected at 4.01 mg/L in the parent sample and at 4.05 mg/L in the duplicate pair; resulting in a RPD of 1.0%. Sulfate was detected at 28.2 mg/L in the parent sample and at 28.0 mg/L in the duplicate pair; resulting in a RPD of 0.7%. Nitrate was detected at 0.290J mg/L in the parent sample and at 0.343J mg/L in the duplicate pair; resulting in a RPD of 16.8%. 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 54MW04 (L13080274-07), sulfate exceeded calibration range. Sample 54MW04 (L13080274-07DL) should be used for this analyte. There were no impacts for data usability based on this outlier.

Sample: 54MW13 (L13080274-01), sulfate

$$Y = mX + b$$

Y = Sample Area

m = slope of curve

X = Concentration (mg/L)

b = Y-intercept

DF = Dilution Factor

Given:

$$m = 0.1379$$

$$b = -0.0209$$

$$Y = \text{Area} = 3.188$$

$$DF = 1$$

$$X = 23.3 \text{ mg/L} \cdot DF = 23.3 \text{ mg/L} \cdot 1 = 23.3 \text{ mg/L}$$

$$\text{Reported concentration} = 23.3 \text{ mg/L}$$

$$\%D = 0.0\%$$

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Certificate of Analysis

Sample #: L13080274-01

PrePrep Method: N/A

Instrument: IC1

Client ID: 54MW13

Prep Method: 9056

Prep Date: 08/06/2013 14:21

Matrix: Water

Analytical Method: 9056

Cal Date: 05/01/2013 13:02

Workgroup #: WG440384

Analyst: KRB

Run Date: 08/06/2013 15:32

Collect Date: 08/05/2013 09:10

Dilution: 1

File ID: I1_080613-07

Sample Tag: 01

Units: mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	2.58		0.200	0.100
Nitrate	14797-55-8	0.591	J J	0.600	0.100
Sulfate	14808-79-8	23.3		1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Certificate of Analysis

Sample #: L13080274-03	PrePrep Method: N/A	Instrument: IC1
Client ID: 54MW01	Prep Method: 9056	Prep Date: 08/06/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 05/01/2013 13:02
Workgroup #: WG440384	Analyst: KRB	Run Date: 08/06/2013 15:50
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: I1_080613-08
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.64		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	26.7		1.00	0.500
U	Analyte was not detected. The concentration is below the reported LOD.				

Certificate of Analysis

Sample #: L13080274-05	PrePrep Method: N/A	Instrument: IC1
Client ID: 54TM01	Prep Method: 9056	Prep Date: 08/06/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 05/01/2013 13:02
Workgroup #: WG440384	Analyst: KRB	Run Date: 08/06/2013 16:08
Collect Date: 08/05/2013 10:40	Dilution: 1	File ID: I1_080613-09
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.67		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.				

Certificate of Analysis

Sample #: L13080274-07

PrePrep Method: N/A

Instrument: IC1

Client ID: 54MW04

Prep Method: 9056

Prep Date: 08/06/2013 14:21

Matrix: Water

Analytical Method: 9056

Cal Date: 05/01/2013 13:02

Workgroup #: WG440384

Analyst: KRB

Run Date: 08/06/2013 16:27

Collect Date: 08/05/2013 12:15

Dilution: 1

File ID: I1_080613-10

Sample Tag: 01

Units: mg/L

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	2.12		0.200	0.100
Nitrate	14797-55-8		U	0.600	0.100
Sulfate	14808-79-8	425	J R	1.00	0.500
J	Estimated value ; the analyte concentration was greater than the highest standard				
U	Analyte was not detected. The concentration is below the reported LOD.				

Certificate of Analysis

Sample #: L13080274-07	PrePrep Method: N/A	Instrument: IC1
Client ID: 54MW04	Prep Method: 9056	Prep Date: 08/06/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 05/01/2013 13:02
Workgroup #: WG440384	Analyst: KRB	Run Date: 08/06/2013 23:30
Collect Date: 08/05/2013 12:15	Dilution: 5	File ID: I1_080613-33
Sample Tag: DL01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	1.66		1.00	0.500
Nitrate	14797-55-8		U	3.00	0.500
Sulfate	14808-79-8	322		5.00	2.50
U	Analyte was not detected. The concentration is below the reported LOD.				

Certificate of Analysis

Sample #: L13080274-09	PrePrep Method: N/A	Instrument: IC1
Client ID: 54MW14	Prep Method: 9056	Prep Date: 08/06/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 05/01/2013 13:02
Workgroup #: WG440384	Analyst: KRB	Run Date: 08/06/2013 17:04
Collect Date: 08/05/2013 13:35	Dilution: 1	File ID: I1_080613-12
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.01		0.200	0.100
Nitrate	14797-55-8	0.290	J J	0.600	0.100
Sulfate	14808-79-8	28.2		1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				

Certificate of Analysis

Sample #: L13080274-11	PrePrep Method: N/A	Instrument: IC1
Client ID: 54TM14	Prep Method: 9056	Prep Date: 08/06/2013 14:21
Matrix: Water	Analytical Method: 9056	Cal Date: 05/01/2013 13:02
Workgroup #: WG440384	Analyst: KRB	Run Date: 08/06/2013 17:22
Collect Date: 08/05/2013 13:35	Dilution: 1	File ID: I1_080613-13
Sample Tag: 01	Units: mg/L	

Analyte	CAS #	Result	Qual	LOQ	LOD
Chloride	16887-00-6	4.05		0.200	0.100
Nitrate	14797-55-8	0.343	J J	0.600	0.100
Sulfate	14808-79-8	28.0		1.00	0.500
J	Estimated value ; the analyte concentration was less than the LOQ.				



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon
Test America Laboratories, Inc., SDG 680-101795

DATE: June 25, 2014

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 28, 2014. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 9060. A total of six 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	680-101795-1	54MW13	680-101795-4
54MW10	680-101795-2	54TM12	680-101795-5
54MW12	680-101795-3	54RB052814	680-101795-6

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 Data		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

6/25/14
 Date

**RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG 680-101795**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, 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 05/28/14, the coolers were received on 05/29/14 by the primary laboratory (Test America Laboratories, Inc.) at 4.0°C , 3.8°C , and 11.4°C . The receipt temperature was slightly above criteria for one the coolers containing samples 54MW01 (680-101795-1) and 54MW10 (680-101795-2); therefore, TOC, TIC, DOC, DIC were qualified "L" for detections and "UL" for non-detections for these samples based upon this outlier based upon professional judgment.
- Holding Time Review: The samples were collected on 05/28/14. The TOC, TIC, DOC, and DIC analysis were run on 06/09/14 and 06/10/14. 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, TIC, DOC, and DIC analysis were run on 06/09/14 and 06/10/14. The initial calibration for TC was analyzed on 05/21/14 with a coefficient of determination of 0.9997. 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 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) 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 $\frac{1}{2}$ 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 54RB052814 (680-101795-6) applies to all of the total samples (TOC, TIC) and dissolved samples (DOC and DIC) 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)
06/09/14	TOC	ICB/CCBs	<LOD	NA	None
06/09/14	DOC	ICB/CCBs	<LOD	NA	None
06/10/14	DOC	ICB/CCBs	<LOD	NA	None
06/09/14	TOC	MB 680-333274/43	<1/2MRL	NA	None
06/09/14	DOC	MB 680-333448/3-A	<1/2MRL	NA	None
06/09/14	TOC	54RB052814	<1/2MRL	NA	None
06/09/14	TIC	54RB052814	<1/2MRL	NA	None
06/09/14	DOC	54RB052814	<1/2MRL	NA	None
06/09/14	DIC	54RB052814	<1/2MRL	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 LCSD serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. LCS/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/LCSD results must fall within the control limits (80-120%; RPD≤20%).

- Sample LCS 680-333274/42 was used as the aqueous LCS for TOC analysis on 06/09/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this LCS.
- Samples LCS 680-333448/1-A and LCSD 680-333448/2-A were used as the aqueous LCS/LCSD for DOC analysis on 06/09/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) 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 (80-120%; RPD≤25%).

- Sample 54MW01 (680-101795-1) was used as the aqueous MS/MSD for TOC and DOC analysis on 06/09/14. TOC (79%) and DOC (37%, 35%) were outside criteria. TOC and DOC were qualified "L" for detections and "UL" for non-detections for the spiked sample based upon the low recoveries. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) 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 ($\leq 30\%$ RPD).

- No aqueous laboratory duplicate was analyzed for TIC, DOC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.
- Sample 54MW01 (680-101795-1) was used as the aqueous laboratory duplicate for TOC analysis on 06/09/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this laboratory duplicate.

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 54MW12 (680-101795-3) and 54TM12 (680-101795-5) was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG. All concentrations found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All criteria were met. No qualifiers were applied.

Table 3 Field Precision Analysis Summary for TOC, TIC, DOC, and DIC for Duplicate Pair 54MW12 (680-101795-3) and 54TM12 (680-101795-5)

Compound	Original Sample (mg/L)	Duplicate Pair (mg/L)	%RPD
Total Inorganic Carbon	50	52	3.9
Dissolved Inorganic Carbon	50	46	8.3
Dissolved Organic Carbon	1.0U	1.0U	NA
Total Organic Carbon	0.50U	0.50U	NA

U = Not Detected as <LOD.

LOD = Limit of Detection

NA = Not Applicable

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 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) are presented in **Table 3**. 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	LOD (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW10	TOC/DOC	0.50; 1.0	62	1.0U	NA	J, UJ
54MW10	TIC/DIC	1.0; 1.0	1.7	60	97.2	J
54MW13	TOC/DOC	0.50; 1.0	0.50U	1.1	NA	None
54MW13	TIC/DIC	1.0; 1.0	46	45	NA	None
54RB021914	TOC/DOC	0.50; 1.0	0.50U	1.0U	NA	None
54RB021914	TIC/DIC	1.0; 1.0	1.0U	1.0U	NA	None
54MW01	TOC/DOC	0.50; 1.0	51J	3.7J	NA	None
54MW01	TIC/DIC	1.0; 1.0	1.0U	52	NA	J, UJ
54MW12	TOC/DOC	0.50; 1.0	0.50U	1.0U	NA	None
54MW12	TIC/DIC	1.0; 1.0	50	50	0.0	None
54TM12	TOC/DOC	0.50; 1.0	0.50U	1.0U	NA	None
54TM12	TIC/DIC	1.0; 1.0	52	46	NA	None

J = Estimated value.

U = Not Detected as <LOD.

LOD = Limit of Detection

NA = Not Applicable

Sample: 54MW01 (680-101795-1), TOC

TOC: $Y = m \cdot X \text{ (mg/L)} + b$

$m = 1.831$

$b = 1.012$

$Y = 94.14$

$DF = 1$

$X = (51 \text{ mg/L}) \cdot 1 = 51 \text{ mg/L}$

TOC (mg/L) = 51 mg/L

Reported Value = 51 mg/L

% Difference = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW01

Lab Sample ID: 680-101795-1

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 11:25

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	51	1.0	0.50	mg/L		J L	1	9060A

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW01

Lab Sample ID: 680-101795-1

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 11:25

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	1.0	1.0		mg/L	U	✓	1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW01

Lab Sample ID: 680-101795-1

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 11:25

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	3.7	1.0		mg/L		J L	1	9060
7440-44-0	Dissolved Inorganic Carbon	52	1.0		mg/L		J	1	9060

Form I Copy

1B-IN INORGANIC ANALYSIS DATA SHEET GENERAL CHEMISTRY

Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2
 Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG ID.: 680-101795-01
 Matrix: Water Date Sampled: 05/28/2014 09:40
 Reporting Basis: WET Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	62	1.0	0.50	mg/L		5	1	9060A

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW10

Lab Sample ID: 680-101795-2

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 09:40

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	1.7	1.0		mg/L		J	1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW10

Lab Sample ID: 680-101795-2

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 09:40

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U	VJ	1	9060
7440-44-0	Dissolved Inorganic Carbon	60	1.0		mg/L		J	1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW12

Lab Sample ID: 680-101795-3

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 13:15

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060A

Form I Copy
1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW12 Lab Sample ID: 680-101795-3
Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG ID.: 680-101795-01
Matrix: Water Date Sampled: 05/28/2014 13:15
Reporting Basis: WET Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	50	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW12

Lab Sample ID: 680-101795-3

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 13:15

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	50	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW13

Lab Sample ID: 680-101795-4

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 14:55

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060A

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW13

Lab Sample ID: 680-101795-4

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 14:55

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	46	1.0		mg/L			1	9060

Form I Copy

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW13

Lab Sample ID: 680-101795-4

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 14:55

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.1	1.0		mg/L			1	9060
7440-44-0	Dissolved Inorganic Carbon	45	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54TM12

Lab Sample ID: 680-101795-5

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 13:15

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060A

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54TM12

Lab Sample ID: 680-101795-5

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 13:15

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	52	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54TM12

Lab Sample ID: 680-101795-5

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 13:15

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	46	1.0		mg/L			1	9060

Form I Copy

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54RB052814

Lab Sample ID: 680-101795-6

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 12:25

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060A

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54RB052814

Lab Sample ID: 680-101795-6

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 12:25

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	1.0	1.0		mg/L	U		1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54RB052814

Lab Sample ID: 680-101795-6

Lab Name: TestAmerica Savannah

Job No.: 680-101795-1

SDG ID.: 680-101795-01

Matrix: Water

Date Sampled: 05/28/2014 12:25

Reporting Basis: WET

Date Received: 05/29/2014 10:00

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	1.0	1.0		mg/L	U		1	9060



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Test America Laboratories, Inc., SDG 680-101795

DATE: June 24, 2014

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 28, 2014. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Test America Laboratories, Inc. A total of six 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	680-101795-1	54MW13	680-101795-4
54MW10	680-101795-2	54TM12	680-101795-5
54MW12	680-101795-3	54RB052814	680-101795-6

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 Data		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

6/24/14

 Date

**RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG 680-101795**

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 05/28/14, the coolers were received on 05/29/14 by the primary laboratory (Test America Laboratories, Inc.) at 4.0°C, 3.8°C, and 11.4°C. The receipt temperature was slightly above criteria for one the coolers containing samples 54MW01 (680-101795-1) and 54MW10 (680-101795-2); however, based upon professional judgment did not impact data quality for perchlorate (i.e. non-aromatic). No qualifiers were applied based upon this outlier.
- Holding Time Review: The aqueous samples were collected on 05/28/14 for perchlorate analysis. The aqueous samples were prepped and analyzed on 06/02/14. 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, ¹⁸O-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 $RSD \leq 20\%$ DoD Perchlorate Handbook)
 ICV ($\leq 15\%D$ DoD Perchlorate Handbook)
 CCV/ICS ($\leq 15\%D$ DoD Perchlorate Handbook)
 LODV ($\pm 30\%D$ DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 12/30/13 on instrument LC3062, all criteria were met for all target compounds ($RSD \leq 20\%$). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with coefficients of determination ≥ 0.99 for primary and confirmation columns. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) 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 54RB052814 (680-101795-6) 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)
06/02/14	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
06/02/14	MB 200-72897/4	All perchlorate <½MRL	NA	NA	None
06/02/14	54RB052814	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) $1.0 \pm 2\%$ 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 interference check sample (ICS) verifies inter-element and background correction factors. The ICS is performed at the beginning of each sample analysis run. Control limits are 70-130%.

- All criteria were met for all other runs. 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 LCS 200-72897/5 was used as aqueous LCS for perchlorate analysis dated 06/02/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) 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 (680-101795-1) was used as aqueous MS/MSD for perchlorate analysis dated 06/02/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) 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 groundwater sample duplicate pair 54MW12 (680-101795-3) and 54TM12 (680-101795-5) was analyzed for perchlorate analysis in this SDG. Perchlorate was detected at 1.7 µg/L in the parent sample and at 1.7 µg/L in the duplicate pair; resulting in a RPD of 0.0%. 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: 54MW12 (680-101795-3), 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 = 1.0621

b = -0.001

Y = Area = 185696/101728 = 1.8254

X = 1.72

$$\text{Conc. } \mu\text{g/L} = (\text{Ax} * \text{Cis} * \text{DF})$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard

Cis = Conc. Of internal Standard ($\mu\text{g/L}$)

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (1.72 * 1 * 1) = 1.7 \mu\text{g/L (Signal \#1)}$$

Reported Value = 1.7 $\mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW01 Lab Sample ID: 680-101795-1
Matrix: Water Lab File ID: P060214A06.d
Analysis Method: 6850 Date Collected: 05/28/2014 11:25
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 06/02/2014 12:07
Con. Extract Vol.: _____ Dilution Factor: 1
Injection Volume: 100 (uL) GC Column: IC-Pak AnionH/R ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 72897 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.029	J M J	0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2
Matrix: Water Lab File ID: P060214A09.d
Analysis Method: 6850 Date Collected: 05/28/2014 09:40
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 06/02/2014 12:53
Con. Extract Vol.: _____ Dilution Factor: 1
Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 72897 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.44		0.20	0.019

Form I Copy

FORM I LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW12 Lab Sample ID: 680-101795-3
 Matrix: Water Lab File ID: P060214A10.d
 Analysis Method: 6850 Date Collected: 05/28/2014 13:15
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 06/02/2014 13:08
 Con. Extract Vol.: _____ Dilution Factor: 1
 Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 72897 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	1.7		0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW13 Lab Sample ID: 680-101795-4
Matrix: Water Lab File ID: P060214A11.d
Analysis Method: 6850 Date Collected: 05/28/2014 14:55
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 06/02/2014 13:23
Con. Extract Vol.: _____ Dilution Factor: 1
Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 72897 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.37		0.20	0.019

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54TM12 Lab Sample ID: 680-101795-5
Matrix: Water Lab File ID: P060214A12.d
Analysis Method: 6850 Date Collected: 05/28/2014 13:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 06/02/2014 13:39
Con. Extract Vol.: _____ Dilution Factor: 1
Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 72897 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	1.7		0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54RB052814 Lab Sample ID: 680-101795-6
Matrix: Water Lab File ID: P060214A13.d
Analysis Method: 6850 Date Collected: 05/28/2014 12:25
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 06/02/2014 13:54
Con. Extract Vol.: _____ Dilution Factor: 1
Injection Volume: 100 (uL) GC Column: IC-Pak AnionH/R ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 72897 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.019	U	0.20	0.019



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Test America Laboratories, Inc., SDG 680-101795

DATE: June 23, 2014

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 28, 2014. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of eleven aqueous samples (includes five confirmatory samples and a rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW01	680-101795-1	54MW13	680-101795-4C
54MW10	680-101795-2	54TM12	680-101795-5
54MW10	680-101795-2C	54TM12	680-101795-5C
54MW12	680-101795-3	54RB052814	680-101795-6
54MW12	680-101795-3C	54RB052814	680-101795-6C
54MW13	680-101795-4		

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 Data		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

6/23/14

Date

**RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG 680-101795**

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 05/28/14, the coolers were received on 05/29/14 by the primary laboratory (Test America Laboratories, Inc.) at 4.0°C, 3.8°C, and 11.4°C. The receipt temperature was slightly above criteria for one the coolers containing samples 54MW01 (680-101795-1) and 54MW10 (680-101795-2); therefore, all target aromatic explosives were qualified "L" for detections and "UL" for non-detections for these samples based upon this outlier.
- Holding Time Review: The aqueous samples were collected on 05/28/14. The samples were extracted on 06/02/14 and were analyzed on 06/05/14 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 54RB052814 (680-101795-6) 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)
06/05/14	MB 200-72922/1-A	All target explosives <½MRL	NA	NA	None
06/05/14	54RB052814	Nitroglycerin	2.7J	13.5	54MW12, 54TM12

J = Estimated value <LOQ and >DL.

LOQ = Limit of Quantitation

MRL = Method Reporting Limit

DL = Detection Limit

NA = Not Applicable

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 $\leq 20\%$.

- For the explosives initial calibration performed on 05/06/14 on instrument CH1208, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this initial calibration.
- For the TNX, DNX, and MNX initial calibration performed on 05/14/14 on instrument CH1208, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this initial calibration.
- For the confirmatory explosives initial calibration performed on 05/09/14 on instrument CH1488, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Confirmatory samples 54MW10 (680-101795-2C), 54MW12 (680-101795-3C), 54MW13 (680-101795-4C), 54TM12 (680-101795-5C), and 54RB052814 (680-101795-6C) apply to this initial calibration.
- For the TNX, DNX, and MNX initial calibration performed on 05/14/14 on instrument CH1488, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Confirmatory samples 54MW10 (680-101795-2C), 54MW12 (680-101795-3C), 54MW13 (680-101795-4C), 54TM12 (680-101795-5C), and 54RB052814 (680-101795-6C) 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 $\pm 20\%$.

- For explosives initial calibration verification performed on 05/06/2014 @18:26 on instrument CH1208, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For TNX, DNX, and MNX initial calibration verification performed on 05/14/2014 @14:55 on instrument CH1208, TNX (30.0%) was outside criteria. For all other target compounds, all criteria were met. No aqueous samples were reported using this initial verification calibration; therefore, no qualifiers were applied based upon this outlier.
- For explosives continuing calibration performed on 06/05/2014 @09:16 on instrument CH1208, all criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) were reported using this continuing calibration.

- For TNX, DNX, and MNX continuing calibration performed on 06/05/2014 @09:54 on instrument CH1208, all criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) were reported using this continuing calibration.
- For explosives continuing calibration performed on 06/05/2014 @17:23 on instrument CH1208, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For TNX, DNX, and MNX continuing calibration performed on 06/05/2014 @18:01 on instrument CH1208, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For confirmatory explosives initial calibration verification performed on 05/09/2014 @14:53 on instrument CH1488, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For confirmatory TNX, DNX, and MNX initial calibration verification performed on 05/14/2014 @20:53 on instrument CH1488, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For confirmatory explosives continuing calibration performed on 06/05/2014 @11:22 on instrument CH1488, all criteria were met. No qualifiers were applied. Confirmatory samples 54MW10 (680-101795-2C), 54MW12 (680-101795-3C), 54MW13 (680-101795-4C), 54TM12 (680-101795-5C), and 54RB052814 (680-101795-6C) were reported using this continuing calibration.
- For confirmatory TNX, DNX, and MNX continuing calibration performed on 06/05/2014 @11:56 on instrument CH1488, all criteria were met. No qualifiers were applied. Confirmatory samples 54MW10 (680-101795-2C), 54MW12 (680-101795-3C), 54MW13 (680-101795-4C), 54TM12 (680-101795-5C), and 54RB052814 (680-101795-6C) were reported using this continuing calibration.
- For confirmatory explosives continuing calibration performed on 06/05/2014 @18:49 on instrument CH1488, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For confirmatory TNX, DNX, and MNX continuing calibration performed on 06/05/2014 @19:23 on instrument CH1488, 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 (75-130%)

- For sample 54MW12 (680-101795-3C), surrogate 1,2-dinitrobenzene (157%) was outside criteria. All detections were qualified estimated bias high "K" and non-detections no qualifier based upon the high recovery.
- For sample 54TM12 (680-101795-5C), surrogate 1,2-dinitrobenzene (162%) was outside criteria. All detections were qualified estimated bias high "K" and non-detections no qualifier based upon the high 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 LCS 200-72922/2-A was used as the aqueous LCS for the explosives analysis on 06/05/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW10 (680-101795-2C), 54MW12 (680-101795-3), 54MW12 (680-101795-3C), 54MW13 (680-101795-4), 54MW13 (680-101795-4C), 54TM12 (680-101795-5), 54TM12 (680-101795-5C), 54RB052814 (680-101795-6), and 54RB052814 (680-101795-6C) apply to this LCS.
- Sample LCS 200-72922/3-A was used as the aqueous LCS for the TNX, DNX, and MNX analysis on 06/05/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW10 (680-101795-2C), 54MW12 (680-101795-3), 54MW12 (680-101795-3C), 54MW13 (680-101795-4), 54MW13 (680-101795-4C), 54TM12 (680-101795-5), 54TM12 (680-101795-5C), 54RB052814 (680-101795-6), and 54RB052814 (680-101795-6C) 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 $\leq 20\%$ RPD.

- Sample 54MW01 (680-101795-1) was used as the aqueous MS/MSD for the explosives analysis on 06/05/14. Target compound TNX (551%, 544%) was outside criteria. All RPD criteria were met. The associated LCS was within criteria for all target compounds (Section VI). Target compound TNX was not detected in the spiked sample; therefore no qualification was required based upon the high recoveries. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW10 (680-101795-2C), 54MW12 (680-101795-3), 54MW12 (680-101795-3C), 54MW13 (680-101795-4), 54MW13 (680-101795-4C), 54TM12 (680-101795-5), 54TM12 (680-101795-5C), 54RB052814 (680-101795-6), and 54RB052814 (680-101795-6C) 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 (680-101795-3) and 54TM12 (680-101795-5) was analyzed for explosives analysis in this SDG. All detected explosives found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All other target analytes were non-detect for the duplicate pair. Target compounds 2,6-dinitrotoluene and 4-nitrotoluene were outside criteria and were qualified estimated "J" for the duplicate pair based upon the high RPDs. These outliers were probably due to the trace compound concentrations detected for the duplicate pair (i.e. <LOQ). For all other target explosives, all criteria were met.

**Table 3 Field Precision Analysis Summary for Explosives for
Duplicate Pair 54MW12 (680-101795-3) and 54TM12 (680-101795-5)**

Compound	Original Sample (µg/L)	Duplicate Pair (µg/L)	%RPD
1,3-Dinitrobenzene	2.3	2.2	4.4
2,4,6-Trinitrotoluene	10	11	9.5
2,4-Dinitrotoluene	0.20J	0.16J	22.2
2,6-Dinitrotoluene	0.73J	0.46J	45.4
2-Amino-4,6-dinitrotoluene	4.7	4.8	2.1
2-Nitrotoluene	0.12J	0.11J	8.7
4-Amino-2,6-dinitrotoluene	3.6	3.6	0.0
4-Nitrotoluene	0.14J	0.24J	52.6
DNX	0.051J	0.056J	9.4
MXN	0.19J	0.16J	17.1
Nitroglycerin	2.3J	1.7J	30.0
RDX	2.0	2.2	9.5
TNX	0.50J	0.57J	13.1

J = Estimated value <LOQ and >DL.

LOQ = Limit of Quantitation

DL = Detection Limit

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).
- The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. The calculation was based calibration factors.
- All positive values must have less than or equal to 40% RPD between the primary and secondary columns.
- For sample 54MW10 (680-101795-2), TNX (76.4%), DNX (115%), MNX (95.0%), 1,3-dinitrobenzene (116%), 2,6-dinitrotoluene (154%), 2,4-dinitrotoluene (96.1%), and 4-nitrotoluene (198%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54MW12 (680-101795-3), TNX (89.6%), DNX (171%), MNX (119%), nitroglycerin (139%), 2,6-dinitrotoluene (115%), 2,4-dinitrotoluene (122%), 2-nitrotoluene (144%), and 4-nitrotoluene (199%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54MW13 (680-101795-4), DNX (67.3%), MNX (100%), RDX (55.3%), 1,3-dinitrobenzene (99.4%), 2,6-dinitrotoluene (179%), and 4-nitrotoluene (199%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54TM12 (680-101795-5), TNX (101%), DNX (169%), MNX (126%), nitroglycerin (151%), 2,6-dinitrotoluene (144%), 2-nitrotoluene (141%), and 4-nitrotoluene (199%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54RB052814 (680-101795-6), nitroglycerin (48.3%) was outside confirmatory criteria; therefore, was qualified estimated "J" for the associated sample based upon this outlier.

Sample: 54MW10 (680-101795-2), 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 = 65960.7852

b = -11738.943

Y = Area = 5492911

$$X = 83.45$$

$$\text{Conc. } \mu\text{g/L} = (A_x * V_t * DF) / (V_s)$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

A_x = Amount of compound being measured ($\mu\text{g/L}$).

V_t = Volume of total extract (mL) from bench sheet.

V_s = Volume of sample extracted (mL).

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (83.45 * 10 * 1) / (500) = 1.7 \mu\text{g/L (Signal \#1)}$$

Reported Value = 1.7 $\mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

²The 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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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW01 Lab Sample ID: 680-101795-1
 Matrix: Water Lab File ID: 79020006.D
 Analysis Method: 8330B Date Collected: 05/28/2014 11:25
 Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
 Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 12:23
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 73108 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.031	U J VL	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.032	U VL	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	0.029	U M VL	0.20	0.029
80251-29-2	DNX	0.029	U VL	0.20	0.029
5755-27-1	MNX	0.018	U VL	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.026	U VL	0.20	0.026
606-20-2	2,6-Dinitrotoluene	0.042	U VL	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	0.016	U VL	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	0.017	U M VL	0.20	0.017
99-99-0	4-Nitrotoluene	0.053	U VL	0.20	0.053
88-72-2	2-Nitrotoluene	0.038	U VL	0.20	0.038
55-63-0	Nitroglycerin	1.3	U	4.0	1.3
121-82-4	RDX	0.026	U M VL	0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	96		75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2
 Matrix: Water Lab File ID: 79020007.D
 Analysis Method: 8330B Date Collected: 05/28/2014 09:40
 Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
 Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 13:01
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 73108 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.19	J J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.96	J J	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	11	L	0.20	0.029
80251-29-2	DNX	0.090	M J J	0.20	0.029
5755-27-1	MNX	0.12	M J J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.038	M J J	0.20	0.026
606-20-2	2,6-Dinitrotoluene	0.092	M J J	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	1.6	M L	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	1.7	L	0.20	0.017
99-99-0	4-Nitrotoluene	0.34	M J J	0.20	0.053
88-72-2	2-Nitrotoluene	0.038	U VL	0.20	0.038
55-63-0	Nitroglycerin	1.3	U	4.0	1.3
121-82-4	RDX	8.7	M L	0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	112		75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2
Matrix: Water Lab File ID: 7884A042.D
Analysis Method: 8330B Date Collected: 05/28/2014 09:40
Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 14:48
Con. Extract Vol.: 10000(uL) Dilution Factor: 1
Injection Volume: 450(uL) GC Column: Biphenyl ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 73109 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.083	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.26	J J	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	10	L	0.20	0.029
80251-29-2	DNX	0.34	J M J	0.20	0.029
5755-27-1	MNX	0.34	J M J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.11	J M J	0.20	0.026
606-20-2	2,6-Dinitrotoluene	0.70	J M J	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	1.4	L	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	1.6	L	0.20	0.017
99-99-0	4-Nitrotoluene	58	J M J	0.20	0.053
121-82-4	RDX	7.5	L	0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	113		75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW12 Lab Sample ID: 680-101795-3
 Matrix: Water Lab File ID: 79020008.D
 Analysis Method: 8330B Date Collected: 05/28/2014 13:15
 Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
 Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 13:38
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 73108 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.50	J J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	2.3	M	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	10	M	0.20	0.029
80251-29-2	DNX	0.051	M J J	0.20	0.029
5755-27-1	MXN	0.19	J J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.20	M J J	0.20	0.026
606-20-2	2,6-Dinitrotoluene	0.73	M J J	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	4.7	M	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	3.6	M	0.20	0.017
99-99-0	4-Nitrotoluene	0.14	J J	0.20	0.053
88-72-2	2-Nitrotoluene	0.12	M J J	0.20	0.038
55-63-0	Nitroglycerin	2.3	M J B	4.0	1.3
121-82-4	RDX	2.0		0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	121	M	75-130

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW12 Lab Sample ID: 680-101795-3
 Matrix: Water Lab File ID: 7884A043.D
 Analysis Method: 8330B Date Collected: 05/28/2014 13:15
 Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
 Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 15:22
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 450(uL) GC Column: Biphenyl ID: 4.6(mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 73109 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.19	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	1.9	K	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	8.0	K	0.20	0.029
80251-29-2	DNX	0.65	J J	0.20	0.029
5755-27-1	MNX	0.75	J M J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.048	J M J	0.20	0.026
606-20-2	2,6-Dinitrotoluene	2.7	J M J	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	4.4	K	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	2.8	K	0.20	0.017
99-99-0	4-Nitrotoluene	77	J M J	0.20	0.053
88-72-2	2-Nitrotoluene	0.75	J M J	0.20	0.038
55-63-0	Nitroglycerin	13	J M B	4.0	1.3
121-82-4	RDX	1.9	K	0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	157	Q	75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW13 Lab Sample ID: 680-101795-4
 Matrix: Water Lab File ID: 79020009.D
 Analysis Method: 8330B Date Collected: 05/28/2014 14:55
 Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
 Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 14:16
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 73108 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.11	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.66	J J	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	5.8		0.20	0.029
80251-29-2	DNX	0.14	M J J	0.20	0.029
5755-27-1	MNX	0.083	M J J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.056	J M J	0.20	0.026
606-20-2	2,6-Dinitrotoluene	0.043	M J J	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	0.86	M	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	0.79		0.20	0.017
99-99-0	4-Nitrotoluene	0.15	J J	0.20	0.053
88-72-2	2-Nitrotoluene	0.038	U M	0.20	0.038
55-63-0	Nitroglycerin	1.3	U M	4.0	1.3
121-82-4	RDX	2.5	M J J	0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	119		75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW13 Lab Sample ID: 680-101795-4
Matrix: Water Lab File ID: 7884A044.D
Analysis Method: 8330B Date Collected: 05/28/2014 14:55
Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 15:57
Con. Extract Vol.: 10000(uL) Dilution Factor: 1
Injection Volume: 450(uL) GC Column: Biphenyl ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 73109 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.080	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.22	J J	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	5.3	M	0.20	0.029
80251-29-2	DNX	0.28	J M J	0.20	0.029
5755-27-1	MNX	0.25	J M J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.075	J M J	0.20	0.026
606-20-2	2,6-Dinitrotoluene	0.78	J M J	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	0.85		0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	0.76		0.20	0.017
99-99-0	4-Nitrotoluene	55	J M J	0.20	0.053
121-82-4	RDX	1.4	J J	0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	118		75-130

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54TM12 Lab Sample ID: 680-101795-5
 Matrix: Water Lab File ID: 79020010.D
 Analysis Method: 8330B Date Collected: 05/28/2014 13:15
 Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
 Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 14:53
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 73108 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.57	M J J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	2.2	M	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	11		0.20	0.029
80251-29-2	DNX	0.056	M J J	0.20	0.029
5755-27-1	MXN	0.16	J J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.16	J M	0.20	0.026
606-20-2	2,6-Dinitrotoluene	0.46	M J J	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	4.8	M	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	3.6		0.20	0.017
99-99-0	4-Nitrotoluene	0.24	J J	0.20	0.053
88-72-2	2-Nitrotoluene	0.11	M J J	0.20	0.038
55-63-0	Nitroglycerin	1.7	M J B	4.0	1.3
121-82-4	RDX	2.2		0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	129	M	75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1

SDG No.: 680-101795-01

Client Sample ID: 54TM12 Lab Sample ID: 680-101795-5

Matrix: Water Lab File ID: 7884A045.D

Analysis Method: 8330B Date Collected: 05/28/2014 13:15

Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58

Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 16:31

Con. Extract Vol.: 10000(uL) Dilution Factor: 1

Injection Volume: 450(uL) GC Column: Biphenyl ID: 4.6(mm)

% Moisture: GPC Cleanup: (Y/N) N

Analysis Batch No.: 73109 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.19	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	1.7	M K	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	8.4	K	0.20	0.029
80251-29-2	DNX	0.67	J J	0.20	0.029
5755-27-1	MNX	0.72	J M J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.21	M K	0.20	0.026
606-20-2	2,6-Dinitrotoluene	2.8	J M J	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	4.3	K	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	2.7	K	0.20	0.017
99-99-0	4-Nitrotoluene	75	J M J	0.20	0.053
88-72-2	2-Nitrotoluene	0.65	J J	0.20	0.038
55-63-0	Nitroglycerin	12	J M B	4.0	1.3
121-82-4	RDX	2.0	K	0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	162	Q	75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54RB052814 Lab Sample ID: 680-101795-6
 Matrix: Water Lab File ID: 79020011.D
 Analysis Method: 8330B Date Collected: 05/28/2014 12:25
 Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
 Sample wt/vol: 500(mL) Date Analyzed: 06/05/2014 15:31
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 73108 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.031	U	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.032	U	0.20	0.032
118-96-7	2,4,6-Trinitrotoluene	0.029	U M	0.20	0.029
80251-29-2	DNX	0.029	U	0.20	0.029
5755-27-1	MXN	0.018	U M	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.026	U	0.20	0.026
606-20-2	2,6-Dinitrotoluene	0.042	U	0.20	0.042
35572-78-2	2-Amino-4,6-dinitrotoluene	0.016	U	0.20	0.016
19406-51-0	4-Amino-2,6-dinitrotoluene	0.017	U M	0.20	0.017
99-99-0	4-Nitrotoluene	0.053	U M	0.20	0.053
88-72-2	2-Nitrotoluene	0.038	U	0.20	0.038
55-63-0	Nitroglycerin	2.7	J M J	4.0	1.3
121-82-4	RDX	0.026	U	0.20	0.026

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	102	M	75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54RB052814 Lab Sample ID: 680-101795-6
Matrix: Water Lab File ID: 7884A046.D
Analysis Method: 8330B Date Collected: 05/28/2014 12:25
Extraction Method: 8330-Prep Date Extracted: 06/02/2014 10:58
Sample wt/vol: 500 (mL) Date Analyzed: 06/05/2014 17:06
Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
Injection Volume: 450 (uL) GC Column: Biphenyl ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 73109 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
55-63-0	Nitroglycerin	1.6	J M J	4.0	1.3

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	112		75-130



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Chlorate, Chlorite, Nitrate, and Sulfate
Test America Laboratories, Inc., SDG 680-101795

DATE: June 23, 2014

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 28, 2014. Samples were analyzed for chlorite and chlorate using USEPA Method 300.1 and for chloride, nitrate, and sulfate using USEPA Method 300.0. A total of six 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	680-101795-1	54MW13	680-101795-4
54MW10	680-101795-2	54TM12	680-101795-5
54MW12	680-101795-3	54RB052814	680-101795-6

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 Data		Parameter
Yes	No	
X		Holding Times and Preservation
	X	Initial and Continuing Calibration
X		Blank Analysis
	X	System Monitoring Compounds
	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

6/23/14

Date

**RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG 680-101795**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 28 days for sulfate, chloride, and chlorate; Cool $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 14 days for chlorite; and Cool to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ with H_2SO_4 to $\text{pH} < 2$ 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 05/28/14, the coolers were received on 05/29/14 by the primary laboratory (Test America Laboratories, Inc.) at 4.0°C , 3.8°C , and 11.4°C . The receipt temperature was slightly above criteria for one the coolers containing samples 54MW01 (680-101795-1) and 54MW10 680-101795-2); therefore, nitrate (short holding time anion) was qualified "L" for detections and "UL" for non-detections for these samples based upon this outlier. The other anions are more stable based upon professional judgment.
- Holding Time Review: Samples were collected on 05/28/14. The samples were analyzed on 05/30/14 for nitrate analysis; on 06/06/14 for chloride and sulfate analysis; on 06/05/14, 06/07/14, and 06/10/14 for chlorite analysis; and on 06/03/14 and 06/06/14 for chlorate analysis, respectively. 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.

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

Chlorite and chlorate: 1 – blank
 5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$; $\text{RSD} \leq 15\%$)
 ICV/CCV ($\leq 15\%$)

- Chloride and sulfate analysis was calibrated on 05/21/14 using linear equation techniques to calculate final calculations. Nitrate analysis was calibrated on 05/23/14 using linear equation techniques to calculate final calculations. Chlorite analysis was calibrated on 06/02/14 using linear regression with final concentrations determined using external calibration factor techniques. Chlorate analysis was calibrated on 06/03/14 and 06/05/14 using linear regression with final concentrations determined using external calibration factor techniques. All coefficients of determinations were $r^2 \geq 0.99$ for chloride, chlorite, chlorate, sulfate, and nitrate for all runs. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) 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 54RB052814 (680-101795-6) 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)
06/06/14	Chloride	ICB/CCBs	<LOD	NA	None
06/06/14	Sulfate	ICB/CCBs	<LOD	NA	None
05/30/14	Nitrate	ICB/CCBs	<LOD	NA	None
06/06/14	Chloride	MB 680-332771/29	<½MRL	NA	None
06/06/14	Sulfate	MB 680-332771/29	<½MRL	NA	None
05/30/14	Nitrate	MB 680-331461/5	<½MRL	NA	None
05/30/14	Nitrate	MB 680-331570/30	<½MRL	NA	None
06/06/14	Chloride	54RB052814	<½MRL	NA	None
06/06/14	Sulfate	54RB052814	0.40J	2.00	None
05/30/14	Nitrate	54RB052814	0.032J	0.16	54MW01
Analysis Date	Analysis	QC Blank ID	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
06/05/14	Chlorite	ICB/CCBs	<LOD	NA	None
06/07/14	Chlorite	ICB/CCBs	<LOD	NA	None
06/10/14	Chlorite	ICB/CCBs	<LOD	NA	None
06/03/14	Chlorate	ICB/CCBs	<LOD	NA	None
06/06/14	Chlorate	ICB/CCBs	<LOD	NA	None
06/04/14	Chlorite	MB 680-332427/3	<½MRL	NA	None
06/07/14	Chlorite	MB 680-332958/5	<½MRL	NA	None
06/10/14	Chlorite	MB 680-333328/2	<½MRL	NA	None
06/03/14	Chlorate	MB 680-331936/7	<½MRL	NA	None
06/05/14	Chlorate	MB 680-332566/11	<½MRL	NA	None
06/05/14	Chlorite	54RB052814	<½MRL	NA	None
06/03/14	Chlorate	54RB052814	<½MRL	NA	None

LOD = Limit of Detection

MRL = Method Reporting Limit

MDL = Method Detection Limit

NA = Not Applicable

IV-System Monitoring Compound (Surrogates)

Laboratory performance on individual samples is established by means of spiking activities. Surrogates were performed for chlorite analysis. 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.

Chlorite and chlorate: Dichloroacetic acid (DCAA) (90-115%)

- All criteria were met for all reported samples. No qualifiers were applied.

V-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and 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. Per DoD QSM, all LCS/LCSD results must fall within the specified control limits:

Chloride, nitrate, and sulfate: 90-110%; RPD≤30% (DOD QSM = 80-120%; RPD≤20%)
Chlorite and chlorate: 85-115%; RPD≤10% (DOD QSM = None Listed)

- Samples LCS 680-332771/30 and LCSD 680-332771/31 were used as the aqueous LCS/LCSD for chloride and sulfate analysis on 06/06/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this LCS/LCSD for chloride and sulfate.
- Samples LCS 680-316449/6 and LCSD 680-316449/7 were used as the aqueous LCS/LCSD for nitrate analysis on 05/30/14. All criteria were met. No qualifiers were applied. Samples 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this LCS/LCSD.
- Samples LCS 680-331570/31 and LCSD 680-331570/32 were used as the aqueous LCS/LCSD for nitrate analysis on 05/30/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1) and 54MW10 (680-101795-2) apply to this LCS/LCSD.
- Samples LCS 680-332427/5 and LCSD 680-332427/6 were used as the aqueous LCS/LCSD for chlorite analysis on 06/04/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this LCS/LCSD.
- Samples LCS 680-332958/7 and LCSD 680-332958/8 were used as the aqueous LCS/LCSD for chlorite analysis on 06/07/14. All criteria were met. No qualifiers were applied. Sample 54MW13 (680-101795-4) applies to this LCS/LCSD.
- Samples LCS 680-333328/4 and LCSD 680-333328/5 were used as the aqueous LCS/LCSD for chlorite analysis on 06/10/14. All criteria were met. No qualifiers were applied. Sample 54MW01 (680-101795-1) applies to this LCS/LCSD.
- Samples LCS 680-331936/9 and LCSD 680-331936/10 were used as the aqueous LCS/LCSD for chlorate analysis on 06/03/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this LCS/LCSD.
- Samples LCS 680-332566/13 and LCSD 680-332566/14 were used as the aqueous LCS/LCSD for chlorate analysis on 06/06/14. All criteria were met. No qualifiers were applied. Sample 54MW13 (680-101795-4) applies to this LCS/LCSD.

VI-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.

VII-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:

Chloride, nitrate, and sulfate: 90-110%; RPD≤30% (DOD QSM = 80-120%; RPD≤20%)
Chlorite and chlorate: 75-125%; RPD≤10% (DOD QSM = None Listed)

- Sample 54MW01 (680-101795-1) was used as the aqueous MS/MSD for chloride and sulfate analysis on 06/06/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this MS/MSD.
- Sample 54MW01 (680-101795-1) was used as the aqueous MS/MSD for nitrate analysis on 05/30/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this MS/MSD.
- Sample 54MW01 (680-101795-1) was used as the aqueous MS/MSD for chlorite analysis on 06/10/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54MW13 (680-101795-4), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this MS/MSD.
- Sample 54MW01 (680-101795-1) was used as the aqueous MS/MSD for chlorate analysis on 06/03/14. All criteria were met. No qualifiers were applied. Samples 54MW01 (680-101795-1), 54MW10 (680-101795-2), 54MW12 (680-101795-3), 54TM12 (680-101795-5), and 54RB052814 (680-101795-6) apply to this MS/MSD.
- Sample 54MW13 (680-101795-4) was used as the aqueous MS/MSD for chlorate analysis on 06/06/14. All criteria were met. No qualifiers were applied. Sample 54MW13 (680-101795-4) applies 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 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 54MW12 (680-101795-3) and 54TM12 (680-101795-5) was analyzed for anion analysis in this SDG. All detected anions found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All other target anions were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

Table 3 Field Precision Analysis Hits Summary for Anions for Duplicate Pair 54MW12 (680-101795-3) and 54TM12 (680-101795-5)

Compound	Original Sample (mg/L)	Duplicate Pair (mg/L)	%RPD
Chloride	4.7	4.7	0.0
Nitrate as N	1.6	1.6	0.0
Sulfate	31	31	0.0

IX-Quantitation Verification and Data Review

The accuracy of analytical results is verified through the calculation of several parameters. The following calculations were performed for verification.

- The percent difference (%D) between the calculated and the reported values should be within 10%.
- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW01 (680-101795-1), sulfate

$$Y = mX + b$$

Y = Sample Area

m = slope of curve

X = Concentration (mg/L)

b = Y-intercept

DF = Dilution Factor

Given:

$$m = 13671897.9$$

$$b = -2187344.4$$

$$Y = \text{Area} = 397288645$$

$$DF = 1$$

$$X = 29.22 \text{ mg/L} * DF = 29.22 \text{ mg/L} * 1 = 29 \text{ mg/L}$$

Reported concentration = 29 mg/L

$$\%D = 0.0\%$$

Values were within 10% difference.

Sample: LCS 680-332427/5, chlorite

$$\text{Conc. } \mu\text{g/L} = (\text{Amt} * DF * V_t) / (CF * V_o)$$

where: Amt = the response on column (ng/mL) of the sample

CF = Calibration Factor (from initial calibration)

V_t = volume of final extract (mL)

DF = dilution factor

V_o = volume of the sample extracted (mL)

$$\begin{aligned} \text{Conc. } \mu\text{g/L} &= (5397011 \text{ ng/mL} * 1 * 5 \text{ mL}) / (54463.4998 * 5 \text{ mL}) \\ &= 99.1 \text{ ng/mL} = 99.1 \mu\text{g/L} \end{aligned}$$

Reported concentration = 99.1 μg/L

$$\%D = 0.0\%$$

Values were within 10% difference.

Sample: LCS 680-331936/9, chlorate

$$\text{Conc. } \mu\text{g/L} = (\text{Amt} * \text{DF} * \text{Vt}) / (\text{CF} * \text{Vo})$$

where: Amt = the response on column (ng/mL) of the sample
CF = Calibration Factor (from initial calibration)
Vt = volume of final extract (mL)
DF = dilution factor
Vo = volume of the sample extracted (mL)

$$\begin{aligned}\text{Conc. } \mu\text{g/L} &= (2205377 \text{ ng/mL} * 1 * 5 \text{ mL}) / (45621.3311 * 5 \text{ mL}) \\ &= 48.3 \text{ ng/mL} = 48.3 \mu\text{g/L}\end{aligned}$$

Reported concentration = 48.3 $\mu\text{g/L}$

%D = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Form I Copy

FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW01 Lab Sample ID: 680-101795-1
 Matrix: Water Lab File ID: 0606141946-37.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 11:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 06/06/2014 19:46
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 332771 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	29		0.50	0.25
16887-00-6	Chloride	1.4		0.50	0.25

Form I Copy

FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW01 Lab Sample ID: 680-101795-1
 Matrix: Water Lab File ID: 0530141929-33.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 11:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 05/30/2014 19:29
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 331570 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.031	J H B	0.050	0.025

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW01 Lab Sample ID: 680-101795-1
Matrix: Water Lab File ID: 0610141056-48.d
Analysis Method: 300.1B Date Collected: 05/28/2014 11:25
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 06/10/2014 10:56
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 333328 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	98		90-115

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Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW01 Lab Sample ID: 680-101795-1
 Matrix: Water Lab File ID: 0603141633-50.d
 Analysis Method: 300.1B Date Collected: 05/28/2014 11:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 06/03/2014 16:33
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 331936 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14866-68-3	Chlorate	2.1	U	10	2.1

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	106		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2
 Matrix: Water Lab File ID: 0606142048-41.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 09:40
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 06/06/2014 20:48
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 332771 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
16887-00-6	Chloride	3.9		0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2
 Matrix: Water Lab File ID: 0606142032-40.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 09:40
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 06/06/2014 20:32
 Con. Extract Vol.: 1(mL) Dilution Factor: 2
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 332771 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	58		1.0	0.50

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2
Matrix: Water Lab File ID: 0530142015-36.d
Analysis Method: 300.0 Date Collected: 05/28/2014 09:40
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 05/30/2014 20:15
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 25 (uL) GC Column: AS18 ID: 4 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 331570 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.33	H <u>L</u>	0.050	0.025

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2
Matrix: Water Lab File ID: 0605140044-25.d
Analysis Method: 300.1B Date Collected: 05/28/2014 09:40
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 06/05/2014 00:44
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 332427 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	108		90-115

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FORM I

HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1

SDG No.: 680-101795-01

Client Sample ID: 54MW10 Lab Sample ID: 680-101795-2

Matrix: Water Lab File ID: 0603141816-53.d

Analysis Method: 300.1B Date Collected: 05/28/2014 09:40

Extraction Method: _____ Date Extracted: _____

Sample wt/vol: 5(mL) Date Analyzed: 06/03/2014 18:16

Con. Extract Vol.: 5(mL) Dilution Factor: 1

Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)

% Moisture: _____ GPC Cleanup: (Y/N) N

Analysis Batch No.: 331936 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14866-68-3	Chlorate	2.1	U	10	2.1

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	101		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW12 Lab Sample ID: 680-101795-3
 Matrix: Water Lab File ID: 0606142103-42.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 13:15
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 06/06/2014 21:03
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 332771 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	31		0.50	0.25
16887-00-6	Chloride	4.7		0.50	0.25

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FORM I

HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW12 Lab Sample ID: 680-101795-3
Matrix: Water Lab File ID: 0530141151-12.d
Analysis Method: 300.0 Date Collected: 05/28/2014 13:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 05/30/2014 11:51
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 25(uL) GC Column: AS18 ID: 4(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 331461 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	1.6		0.050	0.025

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW12 Lab Sample ID: 680-101795-3
Matrix: Water Lab File ID: 0605140119-26.d
Analysis Method: 300.1B Date Collected: 05/28/2014 13:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 06/05/2014 01:19
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 332427 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	110		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW12 Lab Sample ID: 680-101795-3
 Matrix: Water Lab File ID: 0603141850-54.d
 Analysis Method: 300.1B Date Collected: 05/28/2014 13:15
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 06/03/2014 18:50
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 331936 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14866-68-3	Chlorate	2.1	U	10	2.1

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	101		90-115

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FORM I

HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54MW13 Lab Sample ID: 680-101795-4
Matrix: Water Lab File ID: 0606142118-43.d
Analysis Method: 300.0 Date Collected: 05/28/2014 14:55
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 1(mL) Date Analyzed: 06/06/2014 21:18
Con. Extract Vol.: 1(mL) Dilution Factor: 1
Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 332771 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	27		0.50	0.25
16887-00-6	Chloride	2.2		0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW13 Lab Sample ID: 680-101795-4
 Matrix: Water Lab File ID: 0530141136-11.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 14:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 05/30/2014 11:36
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 25 (uL) GC Column: AS18 ID: 4 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 331461 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.41		0.050	0.025

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW13 Lab Sample ID: 680-101795-4
 Matrix: Water Lab File ID: 0607141810-9.d
 Analysis Method: 300.1B Date Collected: 05/28/2014 14:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 06/07/2014 18:10
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 332958 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	113		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54MW13 Lab Sample ID: 680-101795-4
 Matrix: Water Lab File ID: 0606140613-33.d
 Analysis Method: 300.1B Date Collected: 05/28/2014 14:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 06/06/2014 06:13
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 5(mL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 332566 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14866-68-3	Chlorate	2.1	U	10	2.1

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	100		90-115

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Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54TM12 Lab Sample ID: 680-101795-5
 Matrix: Water Lab File ID: 0606142134-44.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 13:15
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 06/06/2014 21:34
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 332771 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	31		0.50	0.25
16887-00-6	Chloride	4.7		0.50	0.25

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54TM12 Lab Sample ID: 680-101795-5
Matrix: Water Lab File ID: 0530141121-10.d
Analysis Method: 300.0 Date Collected: 05/28/2014 13:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 05/30/2014 11:21
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 25(uL) GC Column: AS18 ID: 4(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 331461 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	1.6		0.050	0.025

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54TM12 Lab Sample ID: 680-101795-5
Matrix: Water Lab File ID: 0605140227-28.d
Analysis Method: 300.1B Date Collected: 05/28/2014 13:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 06/05/2014 02:27
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 332427 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	109		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54TM12 Lab Sample ID: 680-101795-5
 Matrix: Water Lab File ID: 0603141959-56.d
 Analysis Method: 300.1B Date Collected: 05/28/2014 13:15
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 06/03/2014 19:59
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 331936 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14866-68-3	Chlorate	2.1	U	10	2.1

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	99		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54RB052814 Lab Sample ID: 680-101795-6
 Matrix: Water Lab File ID: 0606142149-45.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 12:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 06/06/2014 21:49
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 332771 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	0.40	J <u>J</u>	0.50	0.25
16887-00-6	Chloride	0.25	U	0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
 SDG No.: 680-101795-01
 Client Sample ID: 54RB052814 Lab Sample ID: 680-101795-6
 Matrix: Water Lab File ID: 0530141105-9.d
 Analysis Method: 300.0 Date Collected: 05/28/2014 12:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 05/30/2014 11:05
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 331461 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.032	J 5	0.050	0.025

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54RB052814 Lab Sample ID: 680-101795-6
Matrix: Water Lab File ID: 0605140302-29.d
Analysis Method: 300.1B Date Collected: 05/28/2014 12:25
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 06/05/2014 03:02
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 332427 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	95		90-115

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-101795-1
SDG No.: 680-101795-01
Client Sample ID: 54RB052814 Lab Sample ID: 680-101795-6
Matrix: Water Lab File ID: 0603142034-57.d
Analysis Method: 300.1B Date Collected: 05/28/2014 12:25
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 06/03/2014 20:34
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 331936 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14866-68-3	Chlorate	2.1	U	10	2.1

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	100		90-115



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon
Test America Laboratories, Inc., SDG 680-98791

DATE: June 18, 2014

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, 2014. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 9060. A total of six aqueous samples (includes one rinse blank) were validated. The sample IDs are:


Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW10	680-98791-1	54MW01	680-98791-4
54MW13	680-98791-2	54MW12	680-98791-5
54RB021914	680-98791-3	54TM12	680-98791-6

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 Data		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	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

6/18/14

 Date

**RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG 680-98791**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, 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 02/19/14, the coolers were received on 02/20/14 by the primary laboratory (Test America Laboratories, Inc.) at 3.4°C , 2.4°C , and 5.8°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: The samples were collected on 02/19/14. The TOC, TIC, DOC, and DIC analysis were run on 02/24/14, 02/25/14, and 02/26/14. 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, TIC, DOC, and DIC analysis were run on 02/24/14, 02/25/14, and 02/26/14. The initial calibration for TC was analyzed on 10/03/13 with a coefficient of determination of 0.9998. 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 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) 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 $\frac{1}{2}$ 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 54RB11513 (680-95852-2) applies to all of the total samples (TOC, TIC) and dissolved samples (DOC and DIC) 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)
02/24/14	TOC	ICB/CCBs	<LOD	NA	None
02/25/14	DOC	ICB/CCBs	<LOD	NA	None
02/24/14	TOC	MB 680-317193/2	<1/2MRL	NA	None
02/25/14	DOC	MB 680-317201/2-A	<1/2MRL	NA	None
02/24/14	TOC	54RB11513	<1/2MRL	NA	None
02/25/14	TIC	54RB11513	<1/2MRL	NA	None
02/25/14	DOC	54RB11513	<1/2MRL	NA	None
02/25/14	DIC	54RB11513	<1/2MRL	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. All aqueous LCS results must fall within the control limits (80-120%).

- Sample LCS 680-317193/5 was used as the aqueous LCS for TOC analysis on 02/24/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this LCS.
- Sample LCS 680-317201/1-A was used as the aqueous LCS for DOC analysis on 02/25/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this LCS.

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 (80-120%; RPD≤25%).

- Sample 54MW01 (680-98791-4) was used as the aqueous MS/MSD for TOC and DOC analysis on 02/24/14 and 02/25/14, respectively. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) 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 ($\leq 30\%$ RPD).

- No aqueous laboratory duplicate was analyzed for TOC, TIC, and DIC with this SDG; therefore, it was not evaluated. See Section IV for lab precision statements using LCS/LCSD.
- Sample 54MW10 (680-98791-1) was used as the aqueous laboratory duplicate for DOC analysis on 02/25/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this laboratory duplicate.

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 54MW12 (680-98791-5) and 54TM12 (680-98791-6) was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG. All concentrations found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All criteria were met. No qualifiers were applied.

Table 3 Field Precision Analysis Hits Summary for TOC, TIC, DOC, and DIC for Duplicate Pair 54MW12 (680-98791-5) and 54TM12 (680-98791-6)

Compound	Original Sample (mg/L)	Duplicate Pair (mg/L)	%RPD
Dissolved Inorganic Carbon	70	70	0.0
Dissolved Organic Carbon	1.0U	1.0	NA
Total Inorganic Carbon	81	72	11.8
Total Organic Carbon	1.0	0.50U	NA

J = Estimated value.

U = Not Detected as <LOD.

LOD = Limit of Detection

NA = Not Applicable

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 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) are presented in **Table 3**. 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	LOD (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW10	TOC/DOC	0.50; 1.0	0.53J	1.0U	NA	None
54MW10	TIC/DIC	1.0; 1.0	91	85	NA	None
54MW13	TOC/DOC	0.50; 1.0	0.50U	1.0U	NA	None
54MW13	TIC/DIC	1.0; 1.0	68	68	NA	None
54RB021914	TOC/DOC	0.50; 1.0	0.50U	1.0U	NA	None
54RB021914	TIC/DIC	1.0; 1.0	1.0U	1.0U	NA	None
54MW01	TOC/DOC	0.50; 1.0	1.0	1.0U	NA	None
54MW01	TIC/DIC	1.0; 1.0	59	59	NA	None
54MW12	TOC/DOC	0.50; 1.0	0.50U	1.0	NA	None
54MW12	TIC/DIC	1.0; 1.0	72	70	NA	None
54TM12	TOC/DOC	0.50; 1.0	0.50U	1.0	NA	None
54TM12	TIC/DIC	1.0; 1.0	72	70	NA	None

J = Estimated value.

U = Not Detected as <LOD.

LOD = Limit of Detection

NA = Not Applicable

Sample: 54MW10 (680-98791-1), TOC

$$\text{TOC: } Y = m \cdot X \text{ (mg/L)} + b$$

$$m = 1.761$$

$$b = 0.7001$$

$$Y = 1.628$$

$$DF = 1$$

$$X = (0.53 \text{ mg/L}) \cdot 1 = 0.53 \text{ mg/L}$$

$$\text{TOC (mg/L)} = 0.53 \text{ mg/L}$$

$$\text{Reported Value} = 0.53 \text{ mg/L}$$

$$\% \text{ Difference} = 0.0\%$$

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW10

Lab Sample ID: 680-98791-1

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 10:40

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.53	1.0	0.50	mg/L	J	J	1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW10

Lab Sample ID: 680-98791-1

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 10:40

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	91	1.0		mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW10

Lab Sample ID: 680-98791-1

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 10:40

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	85	1.0		mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW13

Lab Sample ID: 680-98791-2

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 09:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW13

Lab Sample ID: 680-98791-2

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 09:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	68	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW13

Lab Sample ID: 680-98791-2

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 09:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	68	1.0		mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54RB021914

Lab Sample ID: 680-98791-3

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 09:45

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54RB021914

Lab Sample ID: 680-98791-3

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 09:45

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	1.0	1.0		mg/L	U		1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54RB021914

Lab Sample ID: 680-98791-3

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 09:45

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	1.0	1.0		mg/L	U		1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW01

Lab Sample ID: 680-98791-4

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 12:00

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	1.0	1.0	0.50	mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW01

Lab Sample ID: 680-98791-4

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 12:00

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	59	1.0		mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW01

Lab Sample ID: 680-98791-4

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 12:00

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	59	1.0		mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW12

Lab Sample ID: 680-98791-5

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 13:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	1.0	1.0	0.50	mg/L			1	9060

Form I Copy

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW12

Lab Sample ID: 680-98791-5

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 13:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	81	1.0		mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW12

Lab Sample ID: 680-98791-5

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 13:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	70	1.0		mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54TM12

Lab Sample ID: 680-98791-6

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 13:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060

Form I Copy

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54TM12

Lab Sample ID: 680-98791-6

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 13:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	72	1.0		mg/L			1	9060

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54TM12

Lab Sample ID: 680-98791-6

Lab Name: TestAmerica Savannah

Job No.: 680-98791-1

SDG ID.: 680-98791-1

Matrix: Water

Date Sampled: 02/19/2014 13:30

Reporting Basis: WET

Date Received: 02/20/2014 10:19

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L			1	9060
7440-44-0	Dissolved Inorganic Carbon	70	1.0		mg/L			1	9060



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Test America Laboratories, Inc., SDG 680-98791

DATE: June 17, 2014

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, 2014. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Test America Laboratories, Inc. A total of six aqueous samples (includes one rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW10	680-98791-1	54MW01	680-98791-4
54MW13	680-98791-2	54MW12	680-98791-5
54RB021914	680-98791-3	54TM12	680-98791-6

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 Data		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



Date

**RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG 680-98791**

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 02/19/14, the coolers were received on 02/20/14 by the primary laboratory (Test America Laboratories, Inc.) at 3.4°C, 2.4°C, and 5.8°C. All criteria were met. No qualifiers were applied.
- Holding Time Review: The aqueous samples were collected on 02/19/14 for perchlorate analysis. The aqueous samples were prepped and analyzed on 02/26/14. 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, ¹⁸O-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 $RSD \leq 20\%$ DoD Perchlorate Handbook)
 ICV ($\leq 15\%D$ DoD Perchlorate Handbook)
 CCV/ICS ($\leq 15\%D$ DoD Perchlorate Handbook)
 LODV ($\pm 30\%D$ DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 12/30/13 on instrument LC3062, all criteria were met for all target compounds ($RSD \leq 20\%$). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with coefficients of determination ≥ 0.99 for primary and confirmation columns. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) 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 54RB021914 (680-98791-3) 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)
02/26/14	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
02/26/14	MB 200-68871/4	All perchlorate <½MRL	NA	NA	None
02/26/14	54RB021914	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) $1.0 \pm 2\%$ 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 interference check sample (ICS) verifies inter-element and background correction factors. The ICS is performed at the beginning of each sample analysis run. Control limits are 70-130%.

- All criteria were met for all other runs. 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 LCS 200-68871/5 was used as aqueous LCS for perchlorate analysis dated 02/26/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) 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 (680-98791-4) was used as aqueous MS/MSD for perchlorate analysis dated 02/26/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) 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 groundwater sample duplicate pair 54MW12 (680-98791-5) and 54TM12 (680-98791-6) was analyzed for perchlorate analysis in this SDG. Perchlorate was detected at 4.0 µg/L in the parent sample and at 4.3 µg/L in the duplicate pair; resulting in a RPD of 7.2%. 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: 54MW10 (680-98791-1), 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:

$$m = 1.0621$$

$$b = -0.001$$

$$Y = \text{Area} = 36122/81387 = 0.4438$$

$$X = 0.42$$

$$\text{Conc. } \mu\text{g/L} = (\text{Ax} * \text{Cis} * \text{DF})$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard

Cis = Conc. Of internal Standard ($\mu\text{g/L}$)

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (0.42 * 1 * 1) = 0.42 \mu\text{g/L (Signal \#1)}$$

Reported Value = $0.42 \mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Form I Copy

FORM I LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW10 Lab Sample ID: 680-98791-1
 Matrix: Water Lab File ID: P022614A06.d
 Analysis Method: 6850 Date Collected: 02/19/2014 10:40
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/26/2014 14:09
 Con. Extract Vol.: _____ Dilution Factor: 1
 Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68871 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.42		0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
SDG No.: 680-98791-1
Client Sample ID: 54MW13 Lab Sample ID: 680-98791-2
Matrix: Water Lab File ID: P022614A07.d
Analysis Method: 6850 Date Collected: 02/19/2014 09:30
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 02/26/2014 14:24
Con. Extract Vol.: _____ Dilution Factor: 1
Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 68871 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.32		0.20	0.019

Form I Copy

FORM I LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54RB021914 Lab Sample ID: 680-98791-3
 Matrix: Water Lab File ID: P022614A08.d
 Analysis Method: 6850 Date Collected: 02/19/2014 09:45
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/26/2014 14:39
 Con. Extract Vol.: _____ Dilution Factor: 1
 Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68871 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.019	U	0.20	0.019

Form I Copy

FORM I LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW01 Lab Sample ID: 680-98791-4
 Matrix: Water Lab File ID: P022614A09.d
 Analysis Method: 6850 Date Collected: 02/19/2014 12:00
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 02/26/2014 14:55
 Con. Extract Vol.: _____ Dilution Factor: 1
 Injection Volume: 100 (uL) GC Column: IC-Pak AnionH/R ID: 4.6 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68871 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.019	U M	0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
SDG No.: 680-98791-1
Client Sample ID: 54MW12 Lab Sample ID: 680-98791-5
Matrix: Water Lab File ID: P022614A10.d
Analysis Method: 6850 Date Collected: 02/19/2014 13:30
Extraction Method: Date Extracted:
Sample wt/vol: 5 (mL) Date Analyzed: 02/26/2014 15:10
Con. Extract Vol.: Dilution Factor: 1
Injection Volume: 100 (uL) GC Column: IC-Pak AnionH/R ID: 4.6 (mm)
% Moisture: GPC Cleanup: (Y/N) N
Analysis Batch No.: 68871 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	4.0		0.20	0.019

Form I Copy

FORM I LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54TM12 Lab Sample ID: 680-98791-6
 Matrix: Water Lab File ID: P022614A11.d
 Analysis Method: 6850 Date Collected: 02/19/2014 13:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/26/2014 15:25
 Con. Extract Vol.: _____ Dilution Factor: 1
 Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68871 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	4.3		0.20	0.019



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Test America Laboratories, Inc., SDG 680-98791

DATE: June 17, 2014

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, 2014. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of fifteen aqueous samples (includes four dilution samples, five confirmatory samples, and a rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW10	680-98791-1	54MW12	680-98791-5DL
54MW10	680-98791-1C	54MW12	680-98791-5C
54MW13	680-98791-2	54MW12	680-98791-5CDL
54MW13	680-98791-2C	54TM12	680-98791-6
54RB021914	680-98791-3	54TM12	680-98791-6DL
54RB021914	680-98791-3C	54TM12	680-98791-6C
54MW01	680-98791-4	54TM12	680-98791-6CDL
54MW12	680-98791-5		

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 Data		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



Date

**RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG 680-98791**

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 02/19/14, the coolers were received on 02/20/14 by the primary laboratory (Test America Laboratories, Inc.) at 3.4°C, 2.4°C, and 5.8°C. All criteria were met. No qualifiers were applied.
- Holding Time Review: The aqueous samples were collected on 02/19/14. The samples were extracted on 02/25/14 and were analyzed on 02/25/14, 02/26/14, and 02/27/14 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 54RB021914 (680-98791-3) 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)
02/25/14	MB 200-68794/1-A	All target explosives <½MRL	NA	NA	None
02/25/14	54RB021914	2,4,6-Trinitrotoluene	0.060J	0.30	None
02/25/14	54RB021914	2,4-Dinitrotoluene	0.28	1.40	54MW10, 54MW12, 54TM12
02/25/14	54RB021914	2,6-Dinitrotoluene	0.19J	0.95	None
02/25/14	54RB021914	4-Amino-2,6-dinitrotoluene	0.36J	1.80	54MW13
02/25/14	54RB021914	MNX	0.74J	3.70	54MW10, 54MW12, 54TM12

J = Estimated value <LOQ and >DL.

LOQ = Limit of Quantitation

MRL = Method Reporting Limit

DL = Detection Limit

NA = Not Applicable

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 $\leq 20\%$.

- For the explosives initial calibration performed on 01/25/14 on instrument CH1208, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), 54MW12 (680-98791-5DL), 54TM12 (680-98791-6), and 54TM12 (680-98791-6DL) apply to this initial calibration.
- For the TNX, DNX, and MNX initial calibration performed on 01/26/14 on instrument CH1208, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this initial calibration.
- For the confirmatory explosives initial calibration performed on 01/23/14 on instrument CH1488, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Confirmations for samples 54MW10 (680-98791-1C), 54MW13 (680-98791-2C), 54RB021914 (680-98791-3C), 54MW12 (680-98791-5C), 54MW12 (680-98791-5CDL), 54TM12 (680-98791-6C), and 54TM12 (680-98791-6CDL) apply to this initial calibration.
- For the TNX, DNX, and MNX initial calibration performed on 01/24/14 on instrument CH1488, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Confirmations for samples 54MW10 (680-98791-1C), 54MW13 (680-98791-2C), 54RB021914 (680-98791-3C), 54MW12 (680-98791-5C), and 54TM12 (680-98791-6C) 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 $\pm 20\%$.

- For explosives initial calibration verification performed on 01/26/14 @16:38 on instrument CH1208, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For TNX, DNX, and MNX initial calibration verification performed on 01/26/14 @02:48 on instrument CH1208, TNX (33.9%) was outside criteria. For all other target compounds, all criteria were met. No aqueous samples were reported using this initial verification calibration; therefore, no qualifiers were applied based upon this outlier.
- For explosives continuing calibration performed on 02/25/14 @11:59 on instrument CH1208, all criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) were reported using this continuing calibration.

- For TNX, DNX, and MNX continuing calibration performed on 02/25/14 @12:36 on instrument CH1208, all criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) were reported using this continuing calibration.
- For explosives continuing calibration performed on 02/25/14 @20:42 on instrument CH1208, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For TNX, DNX, and MNX continuing calibration performed on 02/25/14 @21:19 on instrument CH1208, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For explosives continuing calibration performed on 02/27/14 @13:26 on instrument CH1208, all criteria were met. No qualifiers were applied. Samples 54MW12 (680-98791-5DL) and 54TM12 (680-98791-6DL) were reported using this continuing calibration.
- For TNX, DNX, and MNX continuing calibration performed on 02/27/14 @14:03 on instrument CH1208, all criteria were met. No qualifiers were applied. Samples 54MW12 (680-98791-5DL) and 54TM12 (680-98791-6DL) were reported using this continuing calibration.
- For explosives continuing calibration performed on 02/27/14 @23:23 on instrument CH1208, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For TNX, DNX, and MNX continuing calibration performed on 02/28/14 @00:01 on instrument CH1208, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For confirmatory explosives initial calibration verification performed on 01/23/14 @22:12 on instrument CH1488, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For confirmatory TNX, DNX, and MNX initial calibration verification performed on 01/24/14 @02:11 on instrument CH1488, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For confirmatory explosives continuing calibration performed on 02/25/14 @12:52 on instrument CH1488, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For confirmatory explosives continuing calibration performed on 02/25/14 @18:34 on instrument CH1488, all criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1C), 54MW13 (680-98791-2C), 54RB021914 (680-98791-3C), 54MW12 (680-98791-5C), and 54TM12 (680-98791-6C) were reported using this continuing calibration.
- For confirmatory TNX, DNX, and MNX continuing calibration performed on 02/25/14 @19:08 on instrument CH1488, all criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1C), 54MW13 (680-98791-2C), 54RB021914 (680-98791-3C), 54MW12 (680-98791-5C), and 54TM12 (680-98791-6C) were reported using this continuing calibration.
- For confirmatory explosives continuing calibration performed on 02/26/14 @02:33 on instrument CH1488, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.

- For confirmatory TNX, DNX, and MNX continuing calibration performed on 02/26/14 @03:07 on instrument CH1488, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For confirmatory explosives continuing calibration performed on 02/27/14 @12:06 on instrument CH1488, all criteria were met. No qualifiers were applied. Samples 54MW12 (680-98791-5CDL) and 54TM12 (680-98791-6CDL) were reported using this continuing calibration.
- For confirmatory TNX, DNX, and MNX continuing calibration performed on 02/27/14 @12:41 on instrument CH1488, all criteria were met. No qualifiers were applied. Samples 54MW12 (680-98791-5CDL) and 54TM12 (680-98791-6CDL) were reported using this continuing calibration.
- For confirmatory explosives continuing calibration performed on 02/27/14 @21:14 on instrument CH1488, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For confirmatory TNX, DNX, and MNX continuing calibration performed on 02/27/14 @21:48 on instrument CH1488, 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 (75-130%)

- For sample 54MW12 (680-98791-5), surrogate 1,2-dinitrobenzene (135%) was outside criteria. All detections were qualified estimated bias high "K" and non-detections no qualifier based upon the high recovery.
- For sample 54MW12 (680-98791-5C), surrogate 1,2-dinitrobenzene (206%) was outside criteria. All detections were qualified estimated bias high "K" and non-detections no qualifier based upon the high recovery.
- For sample 54MW12 (680-98791-5DL), surrogate 1,2-dinitrobenzene (138%) was outside criteria. The surrogate was diluted out; therefore, no qualification was applied based upon the high recovery.
- For sample 54MW12 (680-98791-5CDL), surrogate 1,2-dinitrobenzene (199%) was outside criteria. The surrogate was diluted out; therefore, no qualification was applied based upon the high recovery.
- For sample 54TM12 (680-98791-6), surrogate 1,2-dinitrobenzene (136%) was outside criteria. All detections were qualified estimated bias high "K" and non-detections no qualifier based upon the high recovery.
- For sample 54TM12 (680-98791-6C), surrogate 1,2-dinitrobenzene (204%) was outside criteria. All detections were qualified estimated bias high "K" and non-detections no qualifier based upon the high recovery.
- For sample 54TM12 (680-98791-6DL), surrogate 1,2-dinitrobenzene (139%) was outside criteria. The surrogate was diluted out; therefore, no qualification was applied based upon the high recovery.
- For sample 54TM12 (680-98791-6CDL), surrogate 1,2-dinitrobenzene (203%) was outside criteria. The surrogate was diluted out; therefore, no qualification was applied based upon the high 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 LCS 200-68794/2-A was used as the aqueous LCS for the explosives analysis on 02/25/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW10 (680-98791-1C), 54MW13 (680-98791-2), 54MW13 (680-98791-2C), 54RB021914 (680-98791-3), 54RB021914 (680-98791-3C), 54MW01 (680-98791-4), 54MW12 (680-98791-5), 54MW12 (680-98791-5DL), 54MW12 (680-98791-5C), 54MW12 (680-98791-5CDL), 54TM12 (680-98791-6), 54TM12 (680-98791-6DL), 54TM12 (680-98791-6C), and 54TM12 (680-98791-6CDL) apply to this LCS.
- Sample LCS 200-68794/3-A was used as the aqueous LCS for the TNX, DNX, and MNX analysis on 02/25/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW10 (680-98791-1C), 54MW13 (680-98791-2), 54MW13 (680-98791-2C), 54RB021914 (680-98791-3), 54RB021914 (680-98791-3C), 54MW01 (680-98791-4), 54MW12 (680-98791-5), 54MW12 (680-98791-5DL), 54MW12 (680-98791-5C), 54MW12 (680-98791-5CDL), 54TM12 (680-98791-6), 54TM12 (680-98791-6DL), 54TM12 (680-98791-6C), and 54TM12 (680-98791-6CDL) 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 $\leq 20\%$ RPD.

- Sample 54MW01 (680-98791-4) was used as the aqueous MS/MSD for the explosives analysis on 02/26/14. Target compound TNX (517%, 526%) was outside criteria. All RPD criteria were met. The associated LCS was within criteria for all target compounds (Section VI). Target compound TNX was not detected in the spiked sample; therefore no qualification was required based upon the high recoveries. Samples 54MW10 (680-98791-1), 54MW10 (680-98791-1C), 54MW13 (680-98791-2), 54MW13 (680-98791-2C), 54RB021914 (680-98791-3), 54RB021914 (680-98791-3C), 54MW01 (680-98791-4), 54MW12 (680-98791-5), 54MW12 (680-98791-5DL), 54MW12 (680-98791-5C), 54MW12 (680-98791-5CDL), 54TM12 (680-98791-6), 54TM12 (680-98791-6DL), 54TM12 (680-98791-6C), and 54TM12 (680-98791-6CDL) 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 (680-98791-5) and 54TM12 (680-98791-6) was analyzed for explosives analysis in this SDG. If required, the diluted samples were used in the field duplicate analysis. All detected explosives found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All other target analytes were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

**Table 3 Field Precision Analysis Summary for Explosives for
Duplicate Pair 54MW12 (680-98791-5) and 54TM12 (680-98791-6)**

Compound	Original Sample (µg/L)	Duplicate Pair (µg/L)	%RPD
1,3-Dinitrobenzene	5.3	5.5J	3.7
2,4,6-Trinitrotoluene	29	28	3.5
2,4-Dinitrotoluene	0.31J	0.30J	3.3
2-Amino-4,6-dinitrotoluene	6.4	6.3	1.6
2-Nitrotoluene	3.1J	3.0J	3.3
4-Amino-2,6-dinitrotoluene	3.8	3.8	0.0
DNX	0.36J	0.35J	2.8
MXN	0.31	0.32	3.2
Nitroglycerin	47J	46J	2.2
RDX	8.7	8.6	1.2
TNX	0.47J	0.47J	0.0

J = Estimated value <LOQ and >DL.

LOQ = Limit of Quantitation

DL = Detection Limit

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).
- The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. The calculation was based calibration factors.
- All positive values must have less than or equal to 40% RPD between the primary and secondary columns.
- For sample 54MW10 (680-98791-1), DNX (126%), MNX (57.4%), 1,3-dinitrobenzene (92.2%), and 2-nitrotoluene (147%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54MW13 (680-98791-2), DNX (121%), RDX (56.6%), and 1,3-dinitrobenzene (96.8%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54RB021914 (680-98791-3), MNX (123%), 4-amino-2,6-dinitrotoluene (145%), and 2,6-dinitrotoluene (64.0%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54MW12 (680-98791-5), TNX (48.1%), DNX (104%), nitroglycerin (94.3%), 2,4-dinitrotoluene (130%), and 2-nitrotoluene (135%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54TM12 (680-98791-6), TNX (48.1%), DNX (106%), 1,3-dinitrobenzene (41.2%), nitroglycerin (83.2%), 2,4-dinitrotoluene (130%), and 2-nitrotoluene (135%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.

Sample: 54MW10 (680-98791-1), 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 = 70134.4826

b = -21012.017

Y = Area = 8034773

$$X = 114.86$$

$$\text{Conc. } \mu\text{g/L} = (A_x * V_t * \text{DF}) / (V_s)$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

A_x = Amount of compound being measured ($\mu\text{g/L}$).

V_t = Volume of total extract (mL) from bench sheet.

V_s = Volume of sample extracted (mL).

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (114.86 * 10 * 1) / (500) = 2.3 \mu\text{g/L (Signal \#1)}$$

Reported Value = 2.3 $\mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW10 Lab Sample ID: 680-98791-1
 Matrix: Water Lab File ID: 63150006.D
 Analysis Method: 8330B Date Collected: 02/19/2014 10:40
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500(mL) Date Analyzed: 02/25/2014 15:06
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68808 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.22		0.20	0.031
99-65-0	1,3-Dinitrobenzene	1.4	J M J	0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	13	M	0.20	0.023
80251-29-2	DNX	0.13	J M J	0.20	0.029
5755-27-1	MNX	0.13	J B	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.068	J M B	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.039	U M	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	2.6	M	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	2.3	M	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	1.3	J M J	0.20	0.032
55-63-0	Nitroglycerin	2.4	J M J	4.0	1.4
121-82-4	RDX	5.3		0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	110	M	75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1

SDG No.: 680-98791-1

Client Sample ID: 54MW10 Lab Sample ID: 680-98791-1

Matrix: Water Lab File ID: 63200017.D

Analysis Method: 8330B Date Collected: 02/19/2014 10:40

Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49

Sample wt/vol: 500(mL) Date Analyzed: 02/25/2014 21:25

Con. Extract Vol.: 10000(uL) Dilution Factor: 1

Injection Volume: 450(uL) GC Column: Biphenyl ID: 4.6(mm)

% Moisture: _____ GPC Cleanup:(Y/N) N

Analysis Batch No.: 68817 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.29	M	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.52	J J	0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	12		0.20	0.023
80251-29-2	DNX	0.58	J M J	0.20	0.029
5755-27-1	MNX	0.23	J M B	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.096	J M B	0.20	0.046
35572-78-2	2-Amino-4,6-dinitrotoluene	2.5		0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	2.1		0.20	0.022
88-72-2	2-Nitrotoluene	0.19	J M J	0.20	0.032
55-63-0	Nitroglycerin	2.1	J M J	4.0	1.4
121-82-4	RDX	4.0		0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	124		75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW13 Lab Sample ID: 680-98791-2
 Matrix: Water Lab File ID: 63150008.D
 Analysis Method: 8330B Date Collected: 02/19/2014 09:30
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500 (mL) Date Analyzed: 02/25/2014 16:20
 Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
 Injection Volume: 150 (uL) GC Column: C-18 ID: 4.6 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68808 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.097	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.55	J J	0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	2.8	M	0.20	0.023
80251-29-2	DNX	0.059	J M J	0.20	0.029
5755-27-1	MNX	0.018	U M	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.046	U M	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.039	U M	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	0.67	M	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	0.59	M B	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	0.032	U M	0.20	0.032
55-63-0	Nitroglycerin	1.4	U M	4.0	1.4
121-82-4	RDX	1.6	J J	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	94		75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW13 Lab Sample ID: 680-98791-2
 Matrix: Water Lab File ID: 63200019.D
 Analysis Method: 8330B Date Collected: 02/19/2014 09:30
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500 (mL) Date Analyzed: 02/25/2014 22:34
 Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
 Injection Volume: 450 (uL) GC Column: Biphenyl ID: 4.6 (mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68817 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.14	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.19	J M J	0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	2.5		0.20	0.023
80251-29-2	DNX	0.24	J M J	0.20	0.029
35572-78-2	2-Amino-4,6-dinitrotoluene	0.65		0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	0.52	B	0.20	0.022
121-82-4	RDX	0.88	J M J	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	101		75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54RB021914 Lab Sample ID: 680-98791-3
 Matrix: Water Lab File ID: 63150009.D
 Analysis Method: 8330B Date Collected: 02/19/2014 09:45
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500(mL) Date Analyzed: 02/25/2014 16:58
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68808 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.031	U	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.028	U M	0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	0.058	J M J	0.20	0.023
80251-29-2	DNX	0.029	U	0.20	0.029
5755-27-1	MNX	3.1	J J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.20	M	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.36	J M J	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	0.021	U M	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	0.058	J M J	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U M	0.20	0.058
88-72-2	2-Nitrotoluene	0.032	U	0.20	0.032
55-63-0	Nitroglycerin	1.4	U M	4.0	1.4
121-82-4	RDX	0.044	U	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	98	M	75-130

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54RB021914 Lab Sample ID: 680-98791-3
 Matrix: Water Lab File ID: 63200020.D
 Analysis Method: 8330B Date Collected: 02/19/2014 09:45
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500 (mL) Date Analyzed: 02/25/2014 23:08
 Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
 Injection Volume: 450 (uL) GC Column: Biphenyl ID: 4.6 (mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68817 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
118-96-7	2,4,6-Trinitrotoluene	0.060	J M J	0.20	0.023
5755-27-1	MNX	0.74	J M J	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.28	M	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.19	J M J	0.20	0.039
19406-51-0	4-Amino-2,6-dinitrotoluene	0.36	J J	0.20	0.022

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	111		75-130

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1

SDG No.: 680-98791-1

Client Sample ID: 54MW01 Lab Sample ID: 680-98791-4

Matrix: Water Lab File ID: 63150010.D

Analysis Method: 8330B Date Collected: 02/19/2014 12:00

Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49

Sample wt/vol: 500(mL) Date Analyzed: 02/25/2014 17:35

Con. Extract Vol.: 10000(uL) Dilution Factor: 1

Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)

% Moisture: GPC Cleanup: (Y/N) N

Analysis Batch No.: 68808 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.031	U J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.028	U	0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	0.023	U	0.20	0.023
80251-29-2	DNX	0.029	U	0.20	0.029
5755-27-1	MNX	0.018	U	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.046	U	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.039	U	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	0.021	U	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	0.022	U	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	0.032	U	0.20	0.032
55-63-0	Nitroglycerin	1.4	U	4.0	1.4
121-82-4	RDX	0.044	U	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	99		75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW12 Lab Sample ID: 680-98791-5
 Matrix: Water Lab File ID: 63150011.D
 Analysis Method: 8330B Date Collected: 02/19/2014 13:30
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500 (mL) Date Analyzed: 02/25/2014 18:12
 Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
 Injection Volume: 150 (uL) GC Column: C-18 ID: 4.6 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68808 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.47	J <u>J</u>	0.20	0.031
99-65-0	1,3-Dinitrobenzene	5.3	M <u>K</u>	0.20	0.028
80251-29-2	DNX	0.36	J M <u>J</u>	0.20	0.029
5755-27-1	MXN	0.31	<u>B</u>	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.31	J M <u>B</u>	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.039	U M	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	6.4	M <u>K</u>	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	3.8	M <u>K</u>	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	3.1	J M <u>J</u>	0.20	0.032
55-63-0	Nitroglycerin	47	J M <u>J</u>	4.0	1.4
121-82-4	RDX	8.7	<u>K</u>	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	135	M Q	75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW12 Lab Sample ID: 680-98791-5
 Matrix: Water Lab File ID: 63200022.D
 Analysis Method: 8330B Date Collected: 02/19/2014 13:30
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500 (mL) Date Analyzed: 02/26/2014 00:16
 Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
 Injection Volume: 450 (uL) GC Column: Biphenyl ID: 4.6 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68817 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.77	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	3.7	K	0.20	0.028
80251-29-2	DNX	1.1	J M J	0.20	0.029
5755-27-1	MNX	0.39	M B	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.065	J M B	0.20	0.046
35572-78-2	2-Amino-4,6-dinitrotoluene	6.1	K	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	3.0	K	0.20	0.022
88-72-2	2-Nitrotoluene	0.60	J M J	0.20	0.032
55-63-0	Nitroglycerin	17	J M J	4.0	1.4
121-82-4	RDX	6.2	K	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	206	J Q	75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
SDG No.: 680-98791-1
Client Sample ID: 54MW12 DL Lab Sample ID: 680-98791-5 DL
Matrix: Water Lab File ID: 63580003.D
Analysis Method: 8330B Date Collected: 02/19/2014 13:30
Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
Sample wt/vol: 500 (mL) Date Analyzed: 02/27/2014 13:15
Con. Extract Vol.: 10000 (uL) Dilution Factor: 3
Injection Volume: 450 (uL) GC Column: Biphenyl ID: 4.6 (mm)
% Moisture: GPC Cleanup: (Y/N) N
Analysis Batch No.: 68916 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
118-96-7	2,4,6-Trinitrotoluene	29		0.60	0.069

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	199	Q	75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
SDG No.: 680-98791-1
Client Sample ID: 54MW12 DL Lab Sample ID: 680-98791-5 DL
Matrix: Water Lab File ID: 63570003.D
Analysis Method: 8330B Date Collected: 02/19/2014 13:30
Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
Sample wt/vol: 500 (mL) Date Analyzed: 02/27/2014 14:40
Con. Extract Vol.: 10000 (uL) Dilution Factor: 3
Injection Volume: 150 (uL) GC Column: C-18 ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 68915 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
118-96-7	2,4,6-Trinitrotoluene	28	M	0.60	0.069

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	138	M Q	75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54TM12 Lab Sample ID: 680-98791-6
 Matrix: Water Lab File ID: 63150012.D
 Analysis Method: 8330B Date Collected: 02/19/2014 13:30
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500(mL) Date Analyzed: 02/25/2014 18:50
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68808 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.47	J <u>J</u>	0.20	0.031
99-65-0	1,3-Dinitrobenzene	5.5	J M <u>J</u>	0.20	0.028
80251-29-2	DNX	0.35	J M <u>J</u>	0.20	0.029
5755-27-1	MNX	0.32	<u>B</u>	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.30	J M <u>B</u>	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.039	U M	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	6.3	M <u>K</u>	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	3.8	M <u>K</u>	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	3.0	J M <u>J</u>	0.20	0.032
55-63-0	Nitroglycerin	46	J M <u>J</u>	4.0	1.4
121-82-4	RDX	8.6	<u>K</u>	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	136	M Q	75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54TM12 Lab Sample ID: 680-98791-6
 Matrix: Water Lab File ID: 63200023.D
 Analysis Method: 8330B Date Collected: 02/19/2014 13:30
 Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
 Sample wt/vol: 500 (mL) Date Analyzed: 02/26/2014 00:50
 Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
 Injection Volume: 450 (uL) GC Column: Biphenyl ID: 4.6 (mm)
 % Moisture: GPC Cleanup: (Y/N) N
 Analysis Batch No.: 68817 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.76	J M J	0.20	0.031
99-65-0	1,3-Dinitrobenzene	3.6	J J	0.20	0.028
80251-29-2	DNX	1.1	J M J	0.20	0.029
5755-27-1	MNX	0.34	M B	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.063	J M B	0.20	0.046
35572-78-2	2-Amino-4,6-dinitrotoluene	6.1	K	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	3.0	K	0.20	0.022
88-72-2	2-Nitrotoluene	0.59	J M J	0.20	0.032
55-63-0	Nitroglycerin	19	J M J	4.0	1.4
121-82-4	RDX	6.1	K	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	204	J Q	75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
SDG No.: 680-98791-1
Client Sample ID: 54TM12 DL Lab Sample ID: 680-98791-6 DL
Matrix: Water Lab File ID: 63580004.D
Analysis Method: 8330B Date Collected: 02/19/2014 13:30
Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
Sample wt/vol: 500 (mL) Date Analyzed: 02/27/2014 13:49
Con. Extract Vol.: 10000 (uL) Dilution Factor: 3
Injection Volume: 450 (uL) GC Column: Biphenyl ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 68916 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
118-96-7	2,4,6-Trinitrotoluene	28		0.60	0.069

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	203	Q	75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-98791-1
SDG No.: 680-98791-1
Client Sample ID: 54TM12 DL Lab Sample ID: 680-98791-6 DL
Matrix: Water Lab File ID: 63570004.D
Analysis Method: 8330B Date Collected: 02/19/2014 13:30
Extraction Method: 8330-Prep Date Extracted: 02/25/2014 08:49
Sample wt/vol: 500 (mL) Date Analyzed: 02/27/2014 15:18
Con. Extract Vol.: 10000 (uL) Dilution Factor: 3
Injection Volume: 150 (uL) GC Column: C-18 ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 68915 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
118-96-7	2,4,6-Trinitrotoluene	28	M	0.60	0.069

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	139	M Q	75-130



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Chlorate, Chlorite, Nitrate, and Sulfate
Test America Laboratories, Inc., SDG 680-98791

DATE: June 18, 2014

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, 2014. Samples were analyzed for chlorite and chlorate using USEPA Method 300.1 and for chloride, nitrate, and sulfate using USEPA Method 300.0. A total of six aqueous samples (includes one rinse blank) were validated. The sample IDs are:


Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW10	680-98791-1	54MW01	680-98791-4
54MW13	680-98791-2	54MW12	680-98791-5
54RB021914	680-98791-3	54TM12	680-98791-6

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 Data		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	System Monitoring Compounds
	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.



Eric Malarek, Chemist



Date

**RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG 680-98791**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 28 days for sulfate, chloride, and chlorate; Cool $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 14 days for chlorite; and Cool to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ with H_2SO_4 to $\text{pH} < 2$ 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 02/19/14, the coolers were received on 02/20/14 by the primary laboratory (Test America Laboratories, Inc.) at 3.4°C , 2.4°C , and 5.8°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: Samples were collected on 02/19/14. The samples were analyzed on 02/20/14 for nitrate analysis; on 02/27/14 and 02/28/14 for chloride and sulfate analysis; on 02/22/14 for chlorite analysis; and on 02/28/14 for chlorate analysis, respectively. 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.

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

Chlorite and chlorate: 1 – blank
 5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$; $\text{RSD} \leq 15\%$)
 ICV/CCV ($\leq 15\% \text{D}$)

- Chloride and sulfate analysis was calibrated on 02/27/14 using linear equation techniques to calculate final calculations. Nitrate analysis was calibrated on 01/31/14 using linear equation techniques to calculate final calculations. Chlorite analysis was calibrated on 02/21/14 using linear regression with final concentrations determined using external calibration factor techniques. Chlorate analysis was calibrated on 02/26/14 using linear regression with final concentrations determined using external calibration factor techniques. All coefficients of determinations were $r^2 \geq 0.99$ for chloride, chlorite, chlorate, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) 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 54RB021914 (680-98791-3) 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)
02/27/14	Chloride	ICB/CCBs	<LOD	NA	None
02/27/14	Sulfate	ICB/CCBs	<LOD	NA	None
02/28/14	Sulfate	ICB/CCBs	<LOD	NA	None
02/20/14	Nitrate	ICB/CCBs	<LOD	NA	None
02/27/14	Chloride	MB 680-317560/8	<½MRL	NA	None
02/27/14	Sulfate	MB 680-317560/8	<½MRL	NA	None
02/27/14	Chloride	MB 680-317649/35	<½MRL	NA	None
02/27/14	Sulfate	MB 680-317649/35	<½MRL	NA	None
02/28/14	Sulfate	MB 680-317777/5	<½MRL	NA	None
02/20/14	Nitrate	MB 680-316449/5	<½MRL	NA	None
02/27/14	Chloride	54RB021914	<½MRL	NA	None
02/27/14	Sulfate	54RB021914	<½MRL	NA	None
02/20/14	Nitrate	54RB021914	<½MRL	NA	None
Analysis Date	Analysis	QC Blank ID	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
02/22/14	Chlorite	ICB/CCBs	<LOD	NA	None
02/28/14	Chlorate	ICB/CCBs	<LOD	NA	None
02/22/14	Chlorite	MB 680-316767/11	<½MRL	NA	None
02/28/14	Chlorate	MB 680-317730/26	<½MRL	NA	None
02/22/14	Chlorite	54RB021914	<½MRL	NA	None
02/28/14	Chlorate	54RB021914	<½MRL	NA	None

LOD = Limit of Detection

MRL = Method Reporting Limit

MDL = Method Detection Limit

NA = Not Applicable

IV-System Monitoring Compound (Surrogates)

Laboratory performance on individual samples is established by means of spiking activities. Surrogates were performed for chlorite analysis. 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.

Chlorite and chlorate: Dichloroacetic acid (DCAA) (90-115%)

- All criteria were met for all reported samples. No qualifiers were applied.

V-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and 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. Per DoD QSM, all LCS/LCSD results must fall within the specified control limits:

Chloride, nitrate, and sulfate: 90-110%; RPD≤30% (DOD QSM = 80-120%; RPD≤20%)
Chlorite and chlorate: 85-115%; RPD≤10% (DOD QSM = None Listed)

- Samples LCS 680-317560/9 and LCSD 680-317560/10 were used as the aqueous LCS/LCSD for chloride and sulfate analysis on 02/27/14. All criteria were met. No qualifiers were applied. Samples 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), and 54MW12 (680-98791-5) apply to this LCS/LCSD for chloride and sulfate. Sample 54MW10 (680-98791-1) applies to this LCS/LCSD for chloride.
- Samples LCS 680-317649/36 and LCSD 680-317649/37 were used as the aqueous LCS/LCSD for chloride and sulfate analysis on 02/27/14. All criteria were met. No qualifiers were applied. Sample 54TM12 (680-98791-6) applies to this LCS/LCSD for chloride and sulfate.
- Samples LCS 680-317777/6 and LCSD 680-317777/7 were used as the aqueous LCS/LCSD for sulfate analysis on 02/28/14. All criteria were met. No qualifiers were applied. Sample 54MW10 (680-98791-1) applies to this LCS/LCSD.
- Samples LCS 680-316449/6 and LCSD 680-316449/7 were used as the aqueous LCS/LCSD for nitrate analysis on 02/20/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this LCS/LCSD.
- Samples LCS 680-316767/13 and LCSD 680-316767/14 were used as the aqueous LCS/LCSD for chlorite analysis on 02/22/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this LCS/LCSD.
- Samples LCS 680-317730/28 and LCSD 680-317730/29 were used as the aqueous LCS/LCSD for chlorate analysis on 02/28/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this LCS/LCSD.

VI-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.

VII-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:

Chloride, nitrate, and sulfate: 90-110%; RPD≤30% (DOD QSM = 80-120%; RPD≤20%)
Chlorite and chlorate: 75-125%; RPD≤10% (DOD QSM = None Listed)

- Sample 54MW01 (680-98791-4) was used as the aqueous MS/MSD for chloride and sulfate analysis on 02/27/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this MS/MSD.
- Sample 54MW01 (680-98791-4) was used as the aqueous MS/MSD for nitrate analysis on 02/20/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this MS/MSD.
- Sample 54MW01 (680-98791-4) was used as the aqueous MS/MSD for chlorite analysis on 02/22/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) apply to this MS/MSD.
- Sample 54MW01 (680-98791-4) was used as the aqueous MS/MSD for chlorate analysis on 02/28/14. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-98791-1), 54MW13 (680-98791-2), 54RB021914 (680-98791-3), 54MW01 (680-98791-4), 54MW12 (680-98791-5), and 54TM12 (680-98791-6) 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 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 54MW12 (680-98791-5) and 54TM12 (680-98791-6) was analyzed for anion analysis in this SDG. All detected anions found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All other target anions were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

Table 3 Field Precision Analysis Hits Summary for Anions for Duplicate Pair 54MW12 (680-98791-5) and 54TM12 (680-98791-6)

Compound	Original Sample (mg/L)	Duplicate Pair (mg/L)	%RPD
Chloride	5.6	5.7	1.8
Nitrate as N	3.1	3.1	0.0
Sulfate	36	36	0.0

IX-Quantitation Verification and Data Review

The accuracy of analytical results is verified through the calculation of several parameters. The following calculations were performed for verification.

- The percent difference (%D) between the calculated and the reported values should be within 10%.
- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW10 (680-98791-1), sulfate

$$Y = mX + b$$

Y = Sample Area

m = slope of curve

X = Concentration (mg/L)

b = Y-intercept

DF = Dilution Factor

Given:

$$m = 14210997.1$$

$$b = -2495771.7$$

$$Y = \text{Area} = 629271187$$

$$DF = 2$$

$$X = 44.46 \text{ mg/L} * DF = 44.46 \text{ mg/L} * 2 = 89 \text{ mg/L}$$

Reported concentration = 89 mg/L

$$\%D = 0.0\%$$

Values were within 10% difference.

Sample: LCS 680-316767/13, chlorite

$$\text{Conc. } \mu\text{g/L} = (\text{Amt} * DF * V_t) / (CF * V_o)$$

where: Amt = the response on column (ng/mL) of the sample

CF = Calibration Factor (from initial calibration)

V_t = volume of final extract (mL)

DF = dilution factor

V_o = volume of the sample extracted (mL)

$$\begin{aligned} \text{Conc. } \mu\text{g/L} &= (5899418 \text{ ng/mL} * 1 * 5 \text{ mL}) / (59076.6839 * 5 \text{ mL}) \\ &= 99.9 \text{ ng/mL} = 99.9 \mu\text{g/L} \end{aligned}$$

Reported concentration = 99.9 μg/L

$$\%D = 0.0\%$$

Values were within 10% difference.

Sample: LCS 680-317730/28, chlorate

$$\text{Conc. } \mu\text{g/L} = (\text{Amt} * \text{DF} * \text{Vt}) / (\text{CF} * \text{Vo})$$

where: Amt = the response on column (ng/mL) of the sample
CF = Calibration Factor (from initial calibration)
Vt = volume of final extract (mL)
DF = dilution factor
Vo = volume of the sample extracted (mL)

$$\begin{aligned}\text{Conc. } \mu\text{g/L} &= (2030586 \text{ ng/mL} * 1 * 5 \text{ mL}) / (36466.7219 * 5 \text{ mL}) \\ &= 55.7 \text{ ng/mL} = 55.7 \mu\text{g/L}\end{aligned}$$

Reported concentration = 55.7 $\mu\text{g/L}$

%D = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

²The 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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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW10 Lab Sample ID: 680-98791-1
 Matrix: Water Lab File ID: 0227141840-31.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 10:40
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 02/27/2014 18:40
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317560 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
16887-00-6	Chloride	4.2		0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW10 Lab Sample ID: 680-98791-1
 Matrix: Water Lab File ID: 0228141407-14.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 10:40
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 02/28/2014 14:07
 Con. Extract Vol.: 1(mL) Dilution Factor: 2
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317777 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	89		1.0	0.50

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW10 Lab Sample ID: 680-98791-1
 Matrix: Water Lab File ID: 0220141412-16.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 10:40
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 02/20/2014 14:12
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 25 (uL) GC Column: AS18 ID: 4 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316449 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.47		0.050	0.025

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW10 Lab Sample ID: 680-98791-1
 Matrix: Water Lab File ID: 0222140452-28.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 10:40
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/22/2014 04:52
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316767 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	101		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW10 Lab Sample ID: 680-98791-1
 Matrix: Water Lab File ID: 0228140935-40.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 10:40
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 02/28/2014 09:35
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317730 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW13 Lab Sample ID: 680-98791-2
 Matrix: Water Lab File ID: 0227141855-32.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 09:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 02/27/2014 18:55
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317560 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	31		0.50	0.25
16887-00-6	Chloride	2.2		0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW13 Lab Sample ID: 680-98791-2
 Matrix: Water Lab File ID: 0220141428-17.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 09:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 02/20/2014 14:28
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 25 (uL) GC Column: AS18 ID: 4 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316449 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.44		0.050	0.025

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Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW13 Lab Sample ID: 680-98791-2
 Matrix: Water Lab File ID: 0222140526-29.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 09:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 02/22/2014 05:26
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316767 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	102		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW13 Lab Sample ID: 680-98791-2
 Matrix: Water Lab File ID: 0228141009-41.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 09:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/28/2014 10:09
 Con. Extract Vol.: 5(mL) Dilution Factor: 2
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317730 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	4.2	U	20	4.2

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54RB021914 Lab Sample ID: 680-98791-3
 Matrix: Water Lab File ID: 0227141910-33.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 09:45
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 02/27/2014 19:10
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317560 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	0.25	U	0.50	0.25
16887-00-6	Chloride	0.25	U	0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54RB021914 Lab Sample ID: 680-98791-3
 Matrix: Water Lab File ID: 0220141443-18.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 09:45
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/20/2014 14:43
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316449 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.025	U	0.050	0.025

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Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54RB021914 Lab Sample ID: 680-98791-3
 Matrix: Water Lab File ID: 0222140601-30.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 09:45
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/22/2014 06:01
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316767 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	101		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54RB021914 Lab Sample ID: 680-98791-3
 Matrix: Water Lab File ID: 0228141044-42.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 09:45
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/28/2014 10:44
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317730 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW01 Lab Sample ID: 680-98791-4
 Matrix: Water Lab File ID: 0227141926-34.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 12:00
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 02/27/2014 19:26
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317560 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	30		0.50	0.25
16887-00-6	Chloride	1.4		0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW01 Lab Sample ID: 680-98791-4
 Matrix: Water Lab File ID: 0220141529-21.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 12:00
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/20/2014 15:29
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316449 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.025	U	0.050	0.025

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW01 Lab Sample ID: 680-98791-4
 Matrix: Water Lab File ID: 0222140635-31.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 12:00
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/22/2014 06:35
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316767 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	102		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW01 Lab Sample ID: 680-98791-4
 Matrix: Water Lab File ID: 0228141118-43.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 12:00
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/28/2014 11:18
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317730 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW12 Lab Sample ID: 680-98791-5
 Matrix: Water Lab File ID: 0227142012-37.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 13:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 02/27/2014 20:12
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317560 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	36		0.50	0.25
16887-00-6	Chloride	5.6		0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW12 Lab Sample ID: 680-98791-5
 Matrix: Water Lab File ID: 0220141711-26.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 13:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/20/2014 17:11
 Con. Extract Vol.: 5(mL) Dilution Factor: 4
 Injection Volume: 25(uL) GC Column: AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316449 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	3.1		0.20	0.10

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW12 Lab Sample ID: 680-98791-5
 Matrix: Water Lab File ID: 0222140818-34.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 13:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 02/22/2014 08:18
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316767 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	102		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54MW12 Lab Sample ID: 680-98791-5
 Matrix: Water Lab File ID: 0228141301-46.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 13:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 02/28/2014 13:01
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317730 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54TM12 Lab Sample ID: 680-98791-6
 Matrix: Water Lab File ID: 0227142128-42.d
 Analysis Method: 300.0 Date Collected: 02/19/2014 13:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1(mL) Date Analyzed: 02/27/2014 21:28
 Con. Extract Vol.: 1(mL) Dilution Factor: 1
 Injection Volume: 25(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317649 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	36		0.50	0.25
16887-00-6	Chloride	5.7		0.50	0.25

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
SDG No.: 680-98791-1
Client Sample ID: 54TM12 Lab Sample ID: 680-98791-6
Matrix: Water Lab File ID: 0220141726-27.d
Analysis Method: 300.0 Date Collected: 02/19/2014 13:30
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 02/20/2014 17:26
Con. Extract Vol.: 5(mL) Dilution Factor: 4
Injection Volume: 25(uL) GC Column: AS18 ID: 4(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 316449 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	3.1		0.20	0.10

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54TM12 Lab Sample ID: 680-98791-6
 Matrix: Water Lab File ID: 0222140853-35.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 13:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/22/2014 08:53
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 316767 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	101		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-98791-1
 SDG No.: 680-98791-1
 Client Sample ID: 54TM12 Lab Sample ID: 680-98791-6
 Matrix: Water Lab File ID: 0228141336-47.d
 Analysis Method: 300.1B Date Collected: 02/19/2014 13:30
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 02/28/2014 13:36
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 317730 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Total and Dissolved Organic and Inorganic Carbon
Test America Laboratories, Inc., SDG 680-95852

DATE: June 16, 2014

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 5, 2013. Samples were analyzed for Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Total Inorganic Carbon (TIC), and Dissolved Inorganic Carbon (DIC) using USEPA 9060. A total of six aqueous samples (includes one rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW10	680-95852-1	54MW2	680-95852-4
54RB11513	680-95852-2	54MW12	680-95852-5
54MW13	680-95852-3	54TM12	680-95852-6

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 Data		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	Laboratory Control Sample
	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



Date

**RFAAP VALIDATION REPORT
TOC, TIC, DOC, & DIC REVIEW
SDG 680-95852**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, 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 11/05/13, the coolers were received on 11/06/13 by the primary laboratory (Test America Laboratories, Inc.) at 3.8°C , 4.0°C , and 5.0°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: The samples were collected on 11/05/13. The TOC, TIC, DOC, and DIC analysis were run on 11/09/13. 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, TIC, DOC, and DIC analysis were run on 11/09/13. The initial calibration for TC was analyzed on 10/03/13 with a coefficient of determination of 0.9998. 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 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 $\frac{1}{2}$ 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 54RB11513 (680-95852-2) applies to all of the total samples (TOC, TIC) and dissolved samples (DOC and DIC) 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)
11/09/13	TOC	ICB/CCBs	<LOD	NA	None
11/09/13	DOC	ICB/CCBs	<LOD	NA	None
11/09/13	TOC	MB 680-302559/26	<½MRL	NA	None
11/09/13	DOC	MB 680-302579/2-A	<½MRL	NA	None
11/09/13	TOC	54RB11513	<½MRL	NA	None
11/09/13	TIC	54RB11513	<½MRL	NA	None
11/09/13	DOC	54RB11513	<½MRL	NA	None
11/09/13	DIC	54RB11513	<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. All aqueous LCS results must fall within the control limits (80-120%).

- Sample LCS 680-302559/29 was used as the aqueous LCS for TOC analysis on 11/09/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this LCS.
- Sample LCS 680-302579/1-A was used as the aqueous LCS for DOC analysis on 11/09/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this LCS.

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 (80-120%; RPD≤25%).

- Sample 54MW12 (680-95852-5) was used as the aqueous MS/MSD for TOC and DOC analysis on 11/09/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 groundwater sample duplicate pair 54MW12 (680-95852-5) and 54TM12 (680-95852-6) was analyzed for TOC, TIC, DOC, and DIC analysis in this SDG. All concentrations found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All criteria were met. No qualifiers were applied.

Table 3 Field Precision Analysis Hits Summary for TOC, TIC, DOC, and DIC for Duplicate Pair 54MW12 (680-95852-5) and 54TM12 (680-95852-6)

Compound	Original Sample (mg/L)	Duplicate Pair (mg/L)	%RPD
Dissolved Inorganic Carbon	55	54	1.8
Dissolved Organic Carbon	1.0U	1.0U	NA
Total Inorganic Carbon	58	57	1.7
Total Organic Carbon	0.50J	0.50U	NA

J = Estimated value.

U = Not Detected as <LOD.

LOD = Limit of Detection

NA = Not Applicable

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 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) are presented in **Table 3**. 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	LOD (mg/L)	Total Fraction (mg/L)	Dissolved Fraction (mg/L)	%D	Qualifier
54MW10	TOC/DOC	0.50; 1.0	0.68J	1.0U	NA	None
54MW10	TIC/DIC	1.0; 1.0	70	66	NA	None
54RB11513	TOC/DOC	0.50; 1.0	0.50U	1.0U	NA	None
54RB11513	TIC/DIC	1.0; 1.0	1.0U	1.0U	NA	None
54MW13	TOC/DOC	0.50; 1.0	0.50U	1.0U	NA	None
54MW13	TIC/DIC	1.0; 1.0	80	76	NA	None
54MW2	TOC/DOC	0.50; 1.0	2.5	2.4	NA	None
54MW2	TIC/DIC	1.0; 1.0	75	74	NA	None
54MW12	TOC/DOC	0.50; 1.0	0.50J	1.0U	NA	None
54MW12	TIC/DIC	1.0; 1.0	58	55	NA	None
54TM12	TOC/DOC	0.50; 1.0	0.50U	1.0U	NA	None
54TM12	TIC/DIC	1.0; 1.0	57	54	NA	None

J = Estimated value.

U = Not Detected as <LOD.

LOD = Limit of Detection

NA = Not Applicable

Sample: 54MW10 (680-95852-1), TOC

$$\text{TOC: } Y = m \cdot X \text{ (mg/L)} + b$$

$$m = 1.784$$

$$b = 1.185$$

$$Y = 2.393$$

$$DF = 1$$

$$X = (0.68 \text{ mg/L}) \cdot 1 = 0.68 \text{ mg/L}$$

$$\text{TOC (mg/L)} = 0.68 \text{ mg/L}$$

$$\text{Reported Value} = 0.68 \text{ mg/L}$$

$$\% \text{ Difference} = 0.0\%$$

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW10

Lab Sample ID: 680-95852-1

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 09:55

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.68	1.0	0.50	mg/L	J	J	1	9060

Form I Copy

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW10

Lab Sample ID: 680-95852-1

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 09:55

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	70	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW10

Lab Sample ID: 680-95852-1

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 09:55

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	66	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54RB11513

Lab Sample ID: 680-95852-2

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 09:55

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54RB11513

Lab Sample ID: 680-95852-2

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 09:55

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	1.0	1.0		mg/L	U		1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54RB11513

Lab Sample ID: 680-95852-2

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 09:55

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	1.0	1.0		mg/L	U		1	9060

Form I Copy
1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW13 Lab Sample ID: 680-95852-3
Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG ID.:
Matrix: Water Date Sampled: 11/05/2013 11:25
Reporting Basis: WET Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW13

Lab Sample ID: 680-95852-3

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 11:25

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	80	1.0		mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW13

Lab Sample ID: 680-95852-3

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 11:25

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	76	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW2

Lab Sample ID: 680-95852-4

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 13:00

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	2.5	1.0	0.50	mg/L			1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW2

Lab Sample ID: 680-95852-4

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 13:00

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	75	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW2

Lab Sample ID: 680-95852-4

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 13:00

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	2.4	1.0		mg/L			1	9060
7440-44-0	Dissolved Inorganic Carbon	74	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW12

Lab Sample ID: 680-95852-5

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 14:15

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	J	J	1	9060

Form I Copy

1B-IN

INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54MW12

Lab Sample ID: 680-95852-5

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 14:15

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	58	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54MW12

Lab Sample ID: 680-95852-5

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 14:15

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	55	1.0		mg/L			1	9060

Form I Copy

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54TM12

Lab Sample ID: 680-95852-6

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

Matrix: Water

Date Sampled: 11/05/2013 14:15

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ	DL	Units	C	Q	DIL	Method
7440-44-0	Total Organic Carbon	0.50	1.0	0.50	mg/L	U		1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY

Client Sample ID: 54TM12 Lab Sample ID: 680-95852-6
Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG ID.:
Matrix: Water Date Sampled: 11/05/2013 14:15
Reporting Basis: WET Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
	Total Inorganic Carbon	57	1.0		mg/L			1	9060

1B-IN
INORGANIC ANALYSIS DATA SHEET
GENERAL CHEMISTRY - DISSOLVED

Client Sample ID: 54TM12

Lab Sample ID: 680-95852-6

Lab Name: TestAmerica Savannah

Job No.: 680-95852-1

SDG ID.:

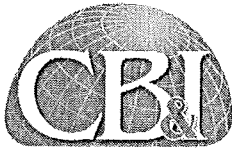
Matrix: Water

Date Sampled: 11/05/2013 14:15

Reporting Basis: WET

Date Received: 11/06/2013 09:37

CAS No.	Analyte	Result	LOQ		Units	C	Q	DIL	Method
7440-44-0	Dissolved Organic Carbon	1.0	1.0		mg/L	U		1	9060
7440-44-0	Dissolved Inorganic Carbon	54	1.0		mg/L			1	9060



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Eric.malarek@CBIfederalservices.com

MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Perchlorate
Test America Laboratories, Inc., SDG 680-95852

DATE: June 13, 2014

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 5, 2013. Aqueous samples were analyzed for perchlorate analysis using liquid chromatography mass spectroscopy (LC/MS) SW-846 method 6850 Modified by Test America Laboratories, Inc. A total of six aqueous samples (includes one rinse blank) were validated. The sample IDs are:


Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW10	680-95852-1	54MW2	680-95852-4
54RB11513	680-95852-2	54MW12	680-95852-5
54MW13	680-95852-3	54TM12	680-95852-6

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 Data		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

6/13/14

 Date

**RFAAP VALIDATION REPORT
PERCHLORATE REVIEW
SDG 680-95852**

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 11/05/13, the coolers were received on 11/06/13 by the primary laboratory (Test America Laboratories, Inc.) at 3.8°C, 4.0°C, and 5.0°C. All criteria were met. No qualifiers were applied.
- Holding Time Review: The aqueous samples were collected on 11/05/13 for perchlorate analysis. The aqueous samples were prepped and analyzed on 11/21/13. 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, ¹⁸O-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 $RSD \leq 20\%$ DoD Perchlorate Handbook)
 ICV ($\leq 15\%D$ DoD Perchlorate Handbook)
 CCV/ICS ($\leq 15\%D$ DoD Perchlorate Handbook)
 LODV ($\pm 30\%D$ DoD Perchlorate Handbook)

- For aqueous perchlorate initial calibration performed on 10/04/13 on instrument LC3062, all criteria were met for all target compounds ($RSD \leq 20\%$). All ICV/CCV/ICS/LODV standards were within criteria. No qualifiers were applied. Perchlorate was determined using linear regression method with coefficients of determination ≥ 0.99 for primary and confirmation columns. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 54RB11513 (680-95852-2) 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)
11/21/13	ICB/CCBs	All perchlorate <½MRL	NA	NA	None
11/21/13	MB200-64811/4	All perchlorate <½MRL	NA	NA	None
11/21/13	54RB11513	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) $1.0 \pm 2\%$ 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 interference check sample (ICS) verifies inter-element and background correction factors. The ICS is performed at the beginning of each sample analysis run. Control limits are 70-130%.

- All criteria were met for all other runs. 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 LCS 200-64811/5 was used as aqueous LCS for perchlorate analysis dated 11/21/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 54MW12 (680-95852-5) was used as aqueous MS/MSD for perchlorate analysis dated 11/21/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 groundwater sample duplicate pair 54MW12 (680-95852-5) and 54TM12 (680-95852-6) was analyzed for perchlorate analysis in this SDG. Perchlorate was detected at 1.1 µg/L in the parent sample and at 1.1 µg/L in the duplicate pair; resulting in a RPD of 0.0%. 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: 54MW10 (680-95852-1), 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 = 1.0098

b = 0.0266

Y = Area = 263947/113480 = 2.326

X = 2.28

$$\text{Conc. } \mu\text{g/L} = (\text{Ax} * \text{Cis} * \text{DF})$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

Ax = Amount Ratio = Conc. Analyte/Conc. Internal Standard

Cis = Conc. Of internal Standard ($\mu\text{g/L}$)

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (2.28 * 1 * 1) = 2.3 \mu\text{g/L (Signal \#1)}$$

Reported Value = $2.3 \mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW10 Lab Sample ID: 680-95852-1
Matrix: Water Lab File ID: P112113B22.d
Analysis Method: 6850 Date Collected: 11/05/2013 09:55
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 11/21/2013 22:02
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 64811 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	2.3		0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54RB11513 Lab Sample ID: 680-95852-2
Matrix: Water Lab File ID: P112113B23.d
Analysis Method: 6850 Date Collected: 11/05/2013 09:55
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 11/21/2013 22:18
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 64811 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.019	U	0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW13 Lab Sample ID: 680-95852-3
Matrix: Water Lab File ID: P112113B24.d
Analysis Method: 6850 Date Collected: 11/05/2013 11:25
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 11/21/2013 22:33
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 100 (uL) GC Column: IC-Pak AnionH/R ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 64811 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.43		0.20	0.019

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW2 Lab Sample ID: 680-95852-4
Matrix: Water Lab File ID: P112113B25.d
Analysis Method: 6850 Date Collected: 11/05/2013 13:00
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 11/21/2013 22:48
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 100 (uL) GC Column: IC-Pak AnionH/R ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 64811 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	0.26		0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW12 Lab Sample ID: 680-95852-5
Matrix: Water Lab File ID: P112113B26.d
Analysis Method: 6850 Date Collected: 11/05/2013 14:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 11/21/2013 23:04
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 100 (uL) GC Column: IC-Pak AnionH/R ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 64811 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	1.1		0.20	0.019

Form I Copy

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54TM12 Lab Sample ID: 680-95852-6
Matrix: Water Lab File ID: P112113B27.d
Analysis Method: 6850 Date Collected: 11/05/2013 14:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 11/21/2013 23:19
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 100(uL) GC Column: IC-Pak AnionH/R ID: 4.6(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 64811 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-73-0	Perchlorate	1.1		0.20	0.019



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Explosives
Test America Laboratories, Inc., SDG 680-95852

DATE: June 13, 2014

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 5, 2013. Samples were analyzed for select explosives using USEPA SW846 Method SW-846 8330B for aqueous matrices. A total of seven aqueous samples (includes one dilution sample and a rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW10	680-95852-1	54MW2	680-95852-4
54MW10	680-95852-1DL	54MW12	680-95852-5
54RB11513	680-95852-2	54TM12	680-95852-6
54MW13	680-95852-3		

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.

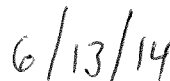
Table 1 Laboratory Performance Criteria

Qualified Data		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



Date

**RFAAP VALIDATION REPORT
EXPLOSIVES REVIEW
SDG 680-95852**

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 11/05/13, the coolers were received on 11/06/13 by the primary laboratory (Test America Laboratories, Inc.) at 3.8°C, 4.0°C, and 5.0°C. All criteria were met. No qualifiers were applied.
- Holding Time Review: The aqueous samples were collected on 11/05/13. The samples were extracted on 11/12/13 and were analyzed on 11/22/13 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 54RB11513 (680-95852-2) 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)
11/21/13	MB 200-64167/1-A	All target explosives <½MRL	NA	NA	None
11/22/13	54RB11513	2,4,6-Trinitrotoluene	0.18J	0.90	None
11/22/13	54RB11513	2,6-Dinitrotoluene	0.32J	1.60	54MW13
11/22/13	54RB11513	4-Amino-2,6-dinitrotoluene	0.077J	0.39	None

J = Estimated value <LOQ and >DL.

LOQ = Limit of Quantitation

MRL = Method Reporting Limit

DL = Detection Limit

NA = Not Applicable

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 $\leq 20\%$.

- For the explosives initial calibration performed on 02/21/13 on instrument CH1208, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this initial calibration.
- For the TNX, DNX, and MNX initial calibration performed on 02/21/13 on instrument CH1208, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this initial calibration.
- For the confirmatory explosives initial calibration performed on 02/21/13 on instrument CH1488, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Confirmations for samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this initial calibration.
- For the TNX, DNX, and MNX initial calibration performed on 02/21/13 on instrument CH1488, all criteria were met for target explosives compounds. All target compounds were determined using linear regression with coefficients of determination ≥ 0.99 . No qualifiers were applied. Confirmations for samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 $\pm 20\%$.

- For explosives initial calibration verification performed on 02/21/13 @23:40 on instrument CH1208, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For TNX, DNX, and MNX initial calibration verification performed on 02/22/13 @04:39 on instrument CH1208, 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 11/21/13 @22:39 on instrument CH1208, all criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) were reported using this continuing calibration.

- For TNX, DNX, and MNX continuing calibration performed on 11/21/13 @23:16 on instrument CH1208, all criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) were reported using this continuing calibration.
- For explosives continuing calibration performed on 11/22/13 @08:37 on instrument CH1208, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For TNX, DNX, and MNX continuing calibration performed on 11/22/13 @09:15 on instrument CH1208, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For confirmatory explosives initial calibration verification performed on 02/21/13 @23:08 on instrument CH1488, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For confirmatory TNX, DNX, and MNX initial calibration verification performed on 02/22/13 @03:41 on instrument CH1488, all criteria were met for target compounds. No qualifiers were applied. No aqueous samples were reported using this initial verification calibration.
- For confirmatory explosives continuing calibration performed on 11/21/13 @21:24 on instrument CH1488, all criteria were met. No qualifiers were applied. Confirmations for samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this continuing calibration.
- For confirmatory TNX, DNX, and MNX continuing calibration performed on 11/21/13 @21:59 on instrument CH1488, all criteria were met. No qualifiers were applied. Confirmations for samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this continuing calibration.
- For confirmatory explosives continuing calibration performed on 11/22/13 @06:31 on instrument CH1488, all criteria were met. No qualifiers were applied. No aqueous samples were reported using this continuing calibration.
- For confirmatory TNX, DNX, and MNX continuing calibration performed on 11/22/13 @07:06 on instrument CH1488, 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 (75-130%)

- All criteria were met for all reported samples. 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 LCS 200-64167/2-A was used as the aqueous LCS for the explosives analysis on 11/22/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this LCS.
- Sample LCS 200-64167/3-A was used as the aqueous LCS for the TNX, DNX, and MNX analysis on 11/22/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 $\leq 20\%$ RPD.

- Sample 54MW12 (680-95852-5) was used as the aqueous MS/MSD for the explosives analysis on 11/22/13. Target compounds TNX (466%, 476%), 1,3-dinitrobenzene (78%, 72%), nitroglycerin (78%), and RDX (129%, 135%) were outside criteria. All RPD criteria were met. The associated LCS was within criteria for all target compounds (Section VI). Target compounds TNX and RDX were detected in the spiked sample; therefore were qualified estimated "K" for detections based upon the high recoveries. Target compounds 1,3-dinitrobenzene and nitroglycerin were qualified estimated "L" for detections and "UL" for non-detections for the spiked sample based upon the low recoveries. Samples 54MW10 (680-95852-1), 54MW10 (680-95852-1DL), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 (680-95852-5) and 54TM12 (680-95852-6) was analyzed for explosives analysis in this SDG. All detected explosives found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All other target analytes were non-detect for the duplicate pair. Target compound 2-nitrotoluene (98.0%) was outside criteria; therefore, was qualified estimated "J" for the duplicate pair based upon the high RPD. The high RPD probably due to low compound concentrations below the LOQ levels.

**Table 3 Field Precision Analysis Summary for Explosives for
Duplicate Pair 54MW12 (680-95852-5) and 54TM12 (680-95852-6)**

Compound	Original Sample (µg/L)	Duplicate Pair (µg/L)	%RPD
1,3-Dinitrobenzene	1.4J	1.4J	0.0
2,4,6-Trinitrotoluene	17	18	5.7
2,4-Dinitrotoluene	0.20J	0.048U	NA
2-Amino-4,6-dinitrotoluene	8.0	8.1	1.2
2-Nitrotoluene	0.19J	0.065J	98.0
4-Amino-2,6-dinitrotoluene	4.4	4.5	2.3
DNX	0.25J	0.25J	0.0
MNX	0.092J	0.096J	4.3
RDX	0.54J	0.51J	5.7
TNX	0.58J	0.59	1.7

J = Estimated value <LOQ and >DL.

LOQ = Limit of Quantitation

DL = Detection Limit

IX-Quantitation Verification and Data Review

The accuracy of analytical results was verified through the calculation of several parameters.

- All values >MDL and <MRL or <3*MDL (whichever was greater) were qualified as estimated "J" (if required).
- The absolute percent difference (%D) between the calculated and the reported value must be within 10% difference. The calculation was based calibration factors.
- All positive values must have less than or equal to 40% RPD between the primary and secondary columns.
- For sample 54MW10 (680-95852-1), TNX (63.9%), DNX (136%), and 1,3-dinitrobenzene (79.6%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54RB11513 (680-95852-2), 2,4,6-trinitrotoluene (72.4%), 4-amino-2,6-dinitrotoluene (46.1%), and 2,6-dinitrotoluene (113%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54MW13 (680-95852-3), TNX (73.6%), DNX (143%), MNX (118%), RDX (64.9%), 1,3-dinitrobenzene (116%), and 2,6-dinitrotoluene (81.6%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54MW12 (680-95852-5), DNX (126%), MNX (118%), RDX (52.0%), 1,3-dinitrobenzene (61.2%), 2,4-dinitrotoluene (116%), and 2-nitrotoluene (49.3%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.
- For sample 54TM12 (680-95852-6), DNX (125%), MNX (71.9%), RDX (53.7%), 1,3-dinitrobenzene (70.0%), and 2-nitrotoluene (138%) were outside confirmatory criteria; therefore, were qualified estimated "J" for the associated sample based upon these outliers.

Sample: 54MW10 (680-95852-1), 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:

$$m = 69089.1333$$

$$b = -31925.667$$

$$Y = \text{Area} = 14830081$$

$$X = 215.11$$

$$\text{Conc. } \mu\text{g/L} = (A_x * V_t * DF) / (V_s)$$

where: Conc. = Sample concentration in $\mu\text{g/L}$

A_x = Amount of compound being measured ($\mu\text{g/L}$).

V_t = Volume of total extract (mL) from bench sheet.

V_s = Volume of sample extracted (mL).

DF = Dilution factor

$$\text{Conc. } \mu\text{g/L} = (215.11 * 10 * 1) / (500) = 4.3 \mu\text{g/L (Signal \#1)}$$

Reported Value = $4.3 \mu\text{g/L}$

% Difference = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

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

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW10 Lab Sample ID: 680-95852-1
 Matrix: Water Lab File ID: PAEGV030.D
 Analysis Method: 8330B Date Collected: 11/05/2013 09:55
 Extraction Method: 8330-Prep Date Extracted: 11/12/2013 09:06
 Sample wt/vol: 500(mL) Date Analyzed: 11/22/2013 02:23
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 64795 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.30	J	J	0.031
99-65-0	1,3-Dinitrobenzene	2.2	M J	J	0.028
80251-29-2	DNX	0.21	J	J	0.029
5755-27-1	MNX	0.41	M		0.018
121-14-2	2,4-Dinitrotoluene	0.20	M		0.046
606-20-2	2,6-Dinitrotoluene	0.039	U		0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	4.5	M		0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	4.3	M		0.022
99-99-0	4-Nitrotoluene	0.058	U		0.058
88-72-2	2-Nitrotoluene	0.032	U		0.032
55-63-0	Nitroglycerin	1.4	U		1.4

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	120		75-130

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW10 DL Lab Sample ID: 680-95852-1 DL
Matrix: Water Lab File ID: PAEGV029.D
Analysis Method: 8330B Date Collected: 11/05/2013 09:55
Extraction Method: 8330-Prep Date Extracted: 11/12/2013 09:06
Sample wt/vol: 500 (mL) Date Analyzed: 11/22/2013 01:46
Con. Extract Vol.: 10000 (uL) Dilution Factor: 3
Injection Volume: 150 (uL) GC Column: C-18 ID: 4.6 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 64795 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
118-96-7	2,4,6-Trinitrotoluene	50	M	0.60	0.069
121-82-4	RDX	21	M	0.60	0.13

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	119		75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54RB11513 Lab Sample ID: 680-95852-2
 Matrix: Water Lab File ID: PAEGV031.D
 Analysis Method: 8330B Date Collected: 11/05/2013 09:55
 Extraction Method: 8330-Prep Date Extracted: 11/12/2013 09:06
 Sample wt/vol: 500(mL) Date Analyzed: 11/22/2013 03:01
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 64795 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.031	U	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.028	U	0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	0.18	M J	J 0.20	0.023
80251-29-2	DNX	0.029	U	0.20	0.029
5755-27-1	MXN	0.018	U M	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.046	U	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.32	M J	J 0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	0.021	U	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	0.077	M J	J 0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	0.032	U M	0.20	0.032
55-63-0	Nitroglycerin	1.4	U	4.0	1.4
121-82-4	RDX	0.044	U	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	102		75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW13 Lab Sample ID: 680-95852-3
 Matrix: Water Lab File ID: PAEGV032.D
 Analysis Method: 8330B Date Collected: 11/05/2013 11:25
 Extraction Method: 8330-Prep Date Extracted: 11/12/2013 09:06
 Sample wt/vol: 500(mL) Date Analyzed: 11/22/2013 03:38
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 64795 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.20	J	J 0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.74	M J	J 0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	9.8	M	0.20	0.023
80251-29-2	DNX	0.17	M J	J 0.20	0.029
5755-27-1	MXN	0.064	J	J 0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.046	U M	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.11	M J	B 0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	1.6	M	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	1.3	M	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	0.032	U	0.20	0.032
55-63-0	Nitroglycerin	1.4	U	4.0	1.4
121-82-4	RDX	4.0	M J	J 0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	113	M	75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW2 Lab Sample ID: 680-95852-4
 Matrix: Water Lab File ID: PAEGV033.D
 Analysis Method: 8330B Date Collected: 11/05/2013 13:00
 Extraction Method: 8330-Prep Date Extracted: 11/12/2013 09:06
 Sample wt/vol: 500 (mL) Date Analyzed: 11/22/2013 04:15
 Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
 Injection Volume: 150 (uL) GC Column: C-18 ID: 4.6 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 64795 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.031	U M	0.20	0.031
99-65-0	1,3-Dinitrobenzene	0.064	J M	0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	0.46	M	0.20	0.023
80251-29-2	DNX	0.029	U	0.20	0.029
5755-27-1	MNX	0.018	U	0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.046	U M	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.039	U	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	0.73	M	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	0.45	M	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	0.032	U	0.20	0.032
55-63-0	Nitroglycerin	1.4	U	4.0	1.4
121-82-4	RDX	0.075	J M	0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	106		75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW12 Lab Sample ID: 680-95852-5
 Matrix: Water Lab File ID: PAEGV035.D
 Analysis Method: 8330B Date Collected: 11/05/2013 14:15
 Extraction Method: 8330-Prep Date Extracted: 11/12/2013 09:06
 Sample wt/vol: 500(mL) Date Analyzed: 11/22/2013 05:30
 Con. Extract Vol.: 10000 (uL) Dilution Factor: 1
 Injection Volume: 150 (uL) GC Column: C-18 ID: 4.6 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 64795 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.58	J M	K 0.20	0.031
99-65-0	1,3-Dinitrobenzene	1.4	J M	L 0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	17	M	0.20	0.023
80251-29-2	DNX	0.25	M J	J 0.20	0.029
5755-27-1	MNX	0.092	J	J 0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.20	M J	J 0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.039	U	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	8.0	M	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	4.4	M	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	0.19	M J	J 0.20	0.032
55-63-0	Nitroglycerin	1.4	U J	UL 4.0	1.4
121-82-4	RDX	0.54	J M	K 0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	124		75-130

Form I Copy

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Burlington Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54TM12 Lab Sample ID: 680-95852-6
 Matrix: Water Lab File ID: PAEGV037.D
 Analysis Method: 8330B Date Collected: 11/05/2013 14:15
 Extraction Method: 8330-Prep Date Extracted: 11/12/2013 09:06
 Sample wt/vol: 500(mL) Date Analyzed: 11/22/2013 06:45
 Con. Extract Vol.: 10000(uL) Dilution Factor: 1
 Injection Volume: 150(uL) GC Column: C-18 ID: 4.6(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 64795 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
13980-04-6	TNX	0.59	M	0.20	0.031
99-65-0	1,3-Dinitrobenzene	1.4	M J	J 0.20	0.028
118-96-7	2,4,6-Trinitrotoluene	18	M	0.20	0.023
80251-29-2	DNX	0.25	M J	J 0.20	0.029
5755-27-1	MXN	0.096	J	J 0.20	0.018
121-14-2	2,4-Dinitrotoluene	0.046	U M	0.20	0.046
606-20-2	2,6-Dinitrotoluene	0.039	U	0.20	0.039
35572-78-2	2-Amino-4,6-dinitrotoluene	8.1	M	0.20	0.021
19406-51-0	4-Amino-2,6-dinitrotoluene	4.5	M	0.20	0.022
99-99-0	4-Nitrotoluene	0.058	U	0.20	0.058
88-72-2	2-Nitrotoluene	0.065	M J	J 0.20	0.032
55-63-0	Nitroglycerin	1.4	U	4.0	1.4
121-82-4	RDX	0.51	M J	J 0.20	0.044

CAS NO.	SURROGATE	%REC	Q	LIMITS
528-29-0	1,2-Dinitrobenzene	133		75-130



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MEMORANDUM

TO: Tim Leahy, CB&I Federal Services, LLC (CB&I) Radford Army Ammunition Plant (RFAAP)
Project Manager

FROM: Eric Malarek, CB&I Project Chemist

SUBJECT: RFAAP Data Validation – Chloride, Chlorate, Chlorite, Nitrate, and Sulfate
Test America Laboratories, Inc., SDG 680-95852

DATE: June 16, 2014

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 5, 2013. Samples were analyzed for chlorite and chlorate using USEPA Method 300.1 and for chloride, nitrate, and sulfate using USEPA Method 300.0. A total of six aqueous samples (includes one rinse blank) were validated. The sample IDs are:

Field Sample ID	Lab Sample ID	Field Sample ID	Lab Sample ID
54MW10	680-95852-1	54MW2	680-95852-4
54RB11513	680-95852-2	54MW12	680-95852-5
54MW13	680-95852-3	54TM12	680-95852-6

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 Data		Parameter
Yes	No	
	X	Holding Times and Preservation
	X	Initial and Continuing Calibration
	X	Blank Analysis
	X	System Monitoring Compounds
	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.



Eric Malarek, Chemist



Date

**RFAAP VALIDATION REPORT
ANIONS REVIEW
SDG 680-95852**

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 28 days for sulfate, chloride, and chlorate; Cool $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and 14 days for chlorite; and Cool to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ with H_2SO_4 to $\text{pH} < 2$ 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 11/05/13, the coolers were received on 11/06/13 by the primary laboratory (Test America Laboratories, Inc.) at 3.8°C , 4.0°C , and 5.0°C . All criteria were met. No qualifiers were applied.
- Holding Time Review: Samples were collected on 11/05/13. The samples were analyzed on 11/06/13 and 11/07/13 for nitrate analysis; on 11/12/13 and 11/13/13 for chloride and sulfate analysis; on 11/9/13 for chlorite analysis; and on 11/15/13 for chlorate analysis, respectively. 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.

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

Chlorite and chlorate: 1 – blank
 5 – standards ($r \geq 0.995$ or $r^2 \geq 0.99$; $\text{RSD} \leq 15\%$)
 ICV/CCV ($\leq 15\% \text{D}$)

- Chloride, sulfate, and nitrate analysis was calibrated on 11/01/13 using linear equation techniques to calculate final calculations. Chlorite analysis was calibrated on 10/27/13 using linear regression with final concentrations determined using external calibration factor techniques. Chlorate analysis was calibrated on 11/13/13 using linear regression with final concentrations determined using external calibration factor techniques. All coefficients of determinations were $r^2 \geq 0.99$ for chloride, chlorite, chlorate, sulfate, and nitrate. All ICV/CCV/MRL criteria were met for all anions and runs. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 54RB11513 (680-95852-2) 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)
11/12/13	Chloride	ICB/CCBs	<LOD	NA	None
11/12/13	Sulfate	ICB/CCBs	<LOD	NA	None
11/13/13	Sulfate	ICB/CCBs	<LOD	NA	None
11/06/13	Nitrate	ICB/CCBs	<LOD	NA	None
11/12/13	Chloride	MB 680-302842/17	<½MRL	NA	None
11/12/13	Sulfate	MB 680-302842/17	<½MRL	NA	None
11/13/13	Sulfate	MB 680-303027/5	<½MRL	NA	None
11/06/13	Nitrate	MB 680-302042/54	<½MRL	NA	None
11/12/13	Chloride	54RB11513	<½MRL	NA	None
11/12/13	Sulfate	54RB11513	<½MRL	NA	None
11/06/13	Nitrate	54RB11513	<½MRL	NA	None
Analysis Date	Analysis	QC Blank ID	Max Conc. µg/L	Action Level µg/L	B qualified samples (for this SDG)
11/09/13	Chlorite	ICB/CCBs	<LOD	NA	None
11/15/13	Chlorate	ICB/CCBs	<LOD	NA	None
11/09/13	Chlorite	MB 680-302151/29	<½MRL	NA	None
11/15/13	Chlorate	MB 680-303452/5	<½MRL	NA	None
11/09/13	Chlorite	54RB11513	<½MRL	NA	None
11/15/13	Chlorate	54RB11513	<½MRL	NA	None

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

IV-System Monitoring Compound (Surrogates)

Laboratory performance on individual samples is established by means of spiking activities. Surrogates were performed for chlorite analysis. 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.

Chlorite and chlorate: Dichloroacetic acid (DCAA) (90-115%)

- All criteria were met for all reported samples. No qualifiers were applied.

V-Laboratory Control Sample and Laboratory Control Sample Duplicate

The laboratory control sample (LCS) and 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. Per DoD QSM, all LCS/LCSD results must fall within the specified control limits:

Chloride, nitrate, and sulfate: 90-110%; RPD≤30% (DOD QSM = 80-120%; RPD≤20%)
Chlorite and chlorate: 85-115%; RPD≤10% (DOD QSM = None Listed)

- Samples LCS 680-302842/18 and LCSD 680-302842/19 were used as the aqueous LCS/LCSD for chloride and sulfate analysis on 11/12/13. All criteria were met. No qualifiers were applied. Samples 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this LCS/LCSD for chloride and sulfate. Sample 54MW10 (680-95852-1) applies to this LCS/LCSD for chloride.
- Samples LCS 680-303027/6 and LCSD 680-303027/7 were used as the aqueous LCS/LCSD for sulfate analysis on 11/13/13. All criteria were met. No qualifiers were applied. Sample 54MW10 (680-95852-1) applies to this LCS/LCSD.
- Samples LCS 680-302042/55 and LCSD 680-302042/56 were used as the aqueous LCS/LCSD for nitrate analysis on 11/06/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this LCS/LCSD.
- Samples LCS 680-302151/31 and LCSD 680-302151/32 were used as the aqueous LCS/LCSD for chlorite analysis on 11/09/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this LCS/LCSD.
- Samples LCS 680-303452/7 and LCSD 680-303452/8 were used as the aqueous LCS/LCSD for chlorate analysis on 11/15/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this LCS/LCSD.

VI-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.

VII-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:

Chloride, nitrate, and sulfate: 90-110%; RPD≤30% (DOD QSM = 80-120%; RPD≤20%)

Chlorite and chlorate: 75-125%; RPD≤10% (DOD QSM = None Listed)

- Sample 54MW12 (680-95852-5) was used as the aqueous MS/MSD for chloride and sulfate analysis on 11/12/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this MS/MSD.
- Sample 54MW12 (680-95852-5) was used as the aqueous MS/MSD for nitrate analysis on 11/06/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this MS/MSD.
- Sample 54MW12 (680-95852-5) was used as the aqueous MS/MSD for chlorite analysis on 11/09/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) apply to this MS/MSD.
- Sample 54MW12 (680-95852-5) was used as the aqueous MS/MSD for chlorate analysis on 11/15/13. All criteria were met. No qualifiers were applied. Samples 54MW10 (680-95852-1), 54RB11513 (680-95852-2), 54MW13 (680-95852-3), 54MW2 (680-95852-4), 54MW12 (680-95852-5), and 54TM12 (680-95852-6) 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 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 54MW12 (680-95852-5) and 54TM12 (680-95852-6) was analyzed for anion analysis in this SDG. All detected anions found in the sample and its duplicate pair and associated %RPD are noted in **Table 3**. All other target anions were non-detect for the duplicate pair. All criteria were met. No qualifiers were applied.

Table 3 Field Precision Analysis Hits Summary for Anions for Duplicate Pair 54MW12 (680-95852-5) and 54TM12 (680-95852-6)

Compound	Original Sample (mg/L)	Duplicate Pair (mg/L)	%RPD
Chloride	5.4	5.4	0.0
Nitrate as N	1.1	1.1	0.0
Sulfate	26	26	0.0

IX-Quantitation Verification and Data Review

The accuracy of analytical results is verified through the calculation of several parameters. The following calculations were performed for verification.

- The percent difference (%D) between the calculated and the reported values should be within 10%.
- Any sample value >MDL and <MRL or <3*MDL (whichever is greater) was qualified as estimated, "J."

Sample: 54MW10 (680-95852-1), sulfate

$$Y = mX + b$$

Y = Sample Area

m = slope of curve

X = Concentration (mg/L)

b = Y-intercept

DF = Dilution Factor

Given:

m = 14523818

b = -46274.385

Y = Area = 412126713

DF = 2

$$X = 28.38 \text{ mg/L} * DF = 28.38 \text{ mg/L} * 2 = 57 \text{ mg/L}$$

Reported concentration = 57 mg/L

%D = 0.0%

Values were within 10% difference.

Sample: LCS 680-302151/31, chlorite

$$\text{Conc. } \mu\text{g/L} = (\text{Amt} * DF * V_t) / (CF * V_o)$$

where: Amt = the response on column (ng/mL) of the sample
CF = Calibration Factor (from initial calibration)
V_t = volume of final extract (mL)
DF = dilution factor
V_o = volume of the sample extracted (mL)

$$\begin{aligned} \text{Conc. } \mu\text{g/L} &= (5737256 \text{ ng/mL} * 1 * 5 \text{ mL}) / (51396.7663 * 5 \text{ mL}) \\ &= 112 \text{ ng/mL} = 112 \mu\text{g/L} \end{aligned}$$

Reported concentration = 112 μg/L

%D = 0.0%

Values were within 10% difference.

Sample: LCS 680-303452/7, chlorate

$$\text{Conc. } \mu\text{g/L} = (\text{Amt} * \text{DF} * \text{Vt}) / (\text{CF} * \text{Vo})$$

where: Amt = the response on column (ng/mL) of the sample
CF = Calibration Factor (from initial calibration)
Vt = volume of final extract (mL)
DF = dilution factor
Vo = volume of the sample extracted (mL)

$$\begin{aligned}\text{Conc. } \mu\text{g/L} &= (2438540 \text{ ng/mL} * 1 * 5 \text{ mL}) / (48884.4503 * 5 \text{ mL}) \\ &= 49.9 \text{ ng/mL} = 49.9 \mu\text{g/L}\end{aligned}$$

Reported concentration = 49.9 $\mu\text{g/L}$

%D = 0.0%

Values were within 10% difference.

Laboratory and Data Validation Qualifiers

Qualifier	Definition
Laboratory Qualifiers¹	
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 Qualifiers²	
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.

¹The 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).

²The 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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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW10 Lab Sample ID: 680-95852-1
 Matrix: Water Lab File ID: 1112131507-21.d
 Analysis Method: 300.0 Date Collected: 11/05/2013 09:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 11/12/2013 15:07
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 1(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 302842 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
16887-00-6	Chloride	4.6	M	0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW10 Lab Sample ID: 680-95852-1
 Matrix: Water Lab File ID: 1113131531-15.d
 Analysis Method: 300.0 Date Collected: 11/05/2013 09:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 11/13/2013 15:31
 Con. Extract Vol.: 5 (mL) Dilution Factor: 2
 Injection Volume: 1 (uL) GC Column: Dionex AS18 ID: 4 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 303027 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	57	M	1.0	0.50

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW10 Lab Sample ID: 680-95852-1
Matrix: Water Lab File ID: 1106132301-64.d
Analysis Method: 300.0 Date Collected: 11/05/2013 09:55
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 11/06/2013 23:01
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 1(uL) GC Column: AS18 ID: 4(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302042 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.67		0.050	0.025

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FORM I

HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica SavannahJob No.: 680-95852-1

SDG No.: _____

Client Sample ID: 54MW10Lab Sample ID: 680-95852-1Matrix: WaterLab File ID: 1109130435-110.dAnalysis Method: 300.1BDate Collected: 11/05/2013 09:55

Extraction Method: _____

Date Extracted: _____

Sample wt/vol: 5(mL)Date Analyzed: 11/09/2013 04:35Con. Extract Vol.: 5(mL)Dilution Factor: 1Injection Volume: 50(uL)GC Column: Dionex AS9-HC ID: 2(mm)

% Moisture: _____

GPC Cleanup: (Y/N) NAnalysis Batch No.: 302151Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	99		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW10 Lab Sample ID: 680-95852-1
 Matrix: Water Lab File ID: 1115131620-11.d
 Analysis Method: 300.1B Date Collected: 11/05/2013 09:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 11/15/2013 16:20
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 303452 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54RB11513 Lab Sample ID: 680-95852-2
 Matrix: Water Lab File ID: 1112131520-22.d
 Analysis Method: 300.0 Date Collected: 11/05/2013 09:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 11/12/2013 15:20
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 1(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 302842 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	0.25	U	0.50	0.25
16887-00-6	Chloride	0.25	U	0.50	0.25

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54RB11513 Lab Sample ID: 680-95852-2
Matrix: Water Lab File ID: 1106132317-65.d
Analysis Method: 300.0 Date Collected: 11/05/2013 09:55
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 11/06/2013 23:17
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 1 (uL) GC Column: AS18 ID: 4 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302042 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.025	U	0.050	0.025

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54RB11513 Lab Sample ID: 680-95852-2
 Matrix: Water Lab File ID: 1109130511-111.d
 Analysis Method: 300.1B Date Collected: 11/05/2013 09:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 11/09/2013 05:11
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 302151 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	101		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54RB11513 Lab Sample ID: 680-95852-2
 Matrix: Water Lab File ID: 1115131656-12.d
 Analysis Method: 300.1B Date Collected: 11/05/2013 09:55
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 11/15/2013 16:56
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 303452 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW13 Lab Sample ID: 680-95852-3
 Matrix: Water Lab File ID: 1112131532-23.d
 Analysis Method: 300.0 Date Collected: 11/05/2013 11:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 11/12/2013 15:32
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 1(uL) GC Column: Dionex AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 302842 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	39	M	0.50	0.25
16887-00-6	Chloride	4.3	M	0.50	0.25

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW13 Lab Sample ID: 680-95852-3
 Matrix: Water Lab File ID: 1106132332-66.d
 Analysis Method: 300.0 Date Collected: 11/05/2013 11:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 11/06/2013 23:32
 Con. Extract Vol.: 5(mL) Dilution Factor: 2
 Injection Volume: 1(uL) GC Column: AS18 ID: 4(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 302042 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.45		0.10	0.050

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW13 Lab Sample ID: 680-95852-3
 Matrix: Water Lab File ID: 1109130547-112.d
 Analysis Method: 300.1B Date Collected: 11/05/2013 11:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 11/09/2013 05:47
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 302151 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	97		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW13 Lab Sample ID: 680-95852-3
 Matrix: Water Lab File ID: 1115131730-13.d
 Analysis Method: 300.1B Date Collected: 11/05/2013 11:25
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 11/15/2013 17:30
 Con. Extract Vol.: 5 (mL) Dilution Factor: 2
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 303452 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	4.2	U	20	4.2

FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW2 Lab Sample ID: 680-95852-4
Matrix: Water Lab File ID: 1112131544-24.d
Analysis Method: 300.0 Date Collected: 11/05/2013 13:00
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 11/12/2013 15:44
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 1(uL) GC Column: Dionex AS18 ID: 4(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302842 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	29	M	0.50	0.25
16887-00-6	Chloride	5.6	M	0.50	0.25

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW2 Lab Sample ID: 680-95852-4
Matrix: Water Lab File ID: 1106132348-67.d
Analysis Method: 300.0 Date Collected: 11/05/2013 13:00
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 11/06/2013 23:48
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 1 (uL) GC Column: AS18 ID: 4 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302042 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	0.25		0.050	0.025

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW2 Lab Sample ID: 680-95852-4
Matrix: Water Lab File ID: 1109130624-113.d
Analysis Method: 300.1B Date Collected: 11/05/2013 13:00
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 11/09/2013 06:24
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302151 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	105		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW2 Lab Sample ID: 680-95852-4
 Matrix: Water Lab File ID: 1115131805-14.d
 Analysis Method: 300.1B Date Collected: 11/05/2013 13:00
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 11/15/2013 18:05
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 303452 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW12 Lab Sample ID: 680-95852-5
Matrix: Water Lab File ID: 1112131557-25.d
Analysis Method: 300.0 Date Collected: 11/05/2013 14:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 11/12/2013 15:57
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 1 (uL) GC Column: Dionex AS18 ID: 4 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302842 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	26	M	0.50	0.25
16887-00-6	Chloride	5.4	M	0.50	0.25

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW12 Lab Sample ID: 680-95852-5
Matrix: Water Lab File ID: 1107130034-70.d
Analysis Method: 300.0 Date Collected: 11/05/2013 14:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 11/07/2013 00:34
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 1(uL) GC Column: AS18 ID: 4(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302042 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	1.1		0.050	0.025

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54MW12 Lab Sample ID: 680-95852-5
Matrix: Water Lab File ID: 1109130700-114.d
Analysis Method: 300.1B Date Collected: 11/05/2013 14:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5(mL) Date Analyzed: 11/09/2013 07:00
Con. Extract Vol.: 5(mL) Dilution Factor: 1
Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302151 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	103		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54MW12 Lab Sample ID: 680-95852-5
 Matrix: Water Lab File ID: 1115131839-15.d
 Analysis Method: 300.1B Date Collected: 11/05/2013 14:15
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 11/15/2013 18:39
 Con. Extract Vol.: 1.0 (mL) Dilution Factor: 1
 Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 303452 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54TM12 Lab Sample ID: 680-95852-6
 Matrix: Water Lab File ID: 1112131634-28.d
 Analysis Method: 300.0 Date Collected: 11/05/2013 14:15
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5 (mL) Date Analyzed: 11/12/2013 16:34
 Con. Extract Vol.: 5 (mL) Dilution Factor: 1
 Injection Volume: 1 (uL) GC Column: Dionex AS18 ID: 4 (mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 302842 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14808-79-8	Sulfate	26	M	0.50	0.25
16887-00-6	Chloride	5.4	M	0.50	0.25

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HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54TM12 Lab Sample ID: 680-95852-6
Matrix: Water Lab File ID: 1107130120-73.d
Analysis Method: 300.0 Date Collected: 11/05/2013 14:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 11/07/2013 01:20
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 1 (uL) GC Column: AS18 ID: 4 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302042 Units: mg/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14797-55-8	Nitrate as N	1.1		0.050	0.025

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FORM I
HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
SDG No.: _____
Client Sample ID: 54TM12 Lab Sample ID: 680-95852-6
Matrix: Water Lab File ID: 1109130849-117.d
Analysis Method: 300.1B Date Collected: 11/05/2013 14:15
Extraction Method: _____ Date Extracted: _____
Sample wt/vol: 5 (mL) Date Analyzed: 11/09/2013 08:49
Con. Extract Vol.: 5 (mL) Dilution Factor: 1
Injection Volume: 50 (uL) GC Column: Dionex AS9-HC ID: 2 (mm)
% Moisture: _____ GPC Cleanup: (Y/N) N
Analysis Batch No.: 302151 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
14998-27-7	Chlorite	3.7	U	20	3.7

CAS NO.	SURROGATE	%REC	Q	LIMITS
79-43-6	Dichloroacetic acid(Surr)	100		90-115

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FORM I HPLC/IC ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-95852-1
 SDG No.: _____
 Client Sample ID: 54TM12 Lab Sample ID: 680-95852-6
 Matrix: Water Lab File ID: 1115132022-18.d
 Analysis Method: 300.1B Date Collected: 11/05/2013 14:15
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 5(mL) Date Analyzed: 11/15/2013 20:22
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 50(uL) GC Column: Dionex AS9-HC ID: 2(mm)
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 303452 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	DL
7790-93-4	Chlorate	2.1	U	10	2.1