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September 1, 2016

William Scruton
QA Coordinator
Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, Minnesota 55155

Re: Quality Assurance Project Plan (QAPP) Addendum #1 – UMore Park/Former Gopher Ordnance Works Remedial Investigation

Dear Mr. Scruton:

On behalf of the University of Minnesota, this letter provides an addendum to the UMore Park/Former Gopher Ordnance Works Remedial Investigation QAPP, dated April 6, 2016. This QAPP addendum addresses updates and additions to the laboratories information in Table 1 and Appendix C as described below:

- Table 1a – updated EPA 6020A method detection limits (MDL) and reporting limits (RL) values. The values originally provided in Table 1a did not account for the initial sample dilution performed on soil samples as part of the laboratory's standard sample preparatory protocol.
- Tables 1a and 1b – added EPA 6010C MDLs, RLs, and quality control limits. Method 6010C will be used for metals (excluding mercury) analysis to minimize future sample dilutions due to background concentrations of certain elements.
- Table 1e – added EPA TO-17 analysis and the associated MDLs, RLs, and quality control limits.
- Appendix C – added Laboratory Standard Operating Procedures for the Determination of Volatile Organic Compounds in Ambient Air Using Active or Passive Sampling onto Sorbent Tubes (ALS), Analysis of Sample by Axial ICP-AES (200.7, 6010C; Legend Technical Services), and Preparation of Aqueous Samples for Testing by ICP (200.7, 6010C; Legend Technical Services).

A copy of the updates will be routed to all recipients after your review and acceptance.

Please contact me by phone (952-832-2763) or email jeidem@barr.com if you have any questions or would like to discuss any of the information above.

Sincerely,

Jim Eidem P.G.
Project Manager

Andrea Nord
Quality Assurance Officer

Attachments

c: Janet Dalgleish, UMN
Kelly Horiuchi, ALS Environmental, Simi Valley, CA
Chaney Humphrey, ALS Environmental, Simi Valley, CA
Ken Kerns, UMN
Gary Krueger, MPCA
Ed Olson, MPCA
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Table 1a
Analytical Parameters, Methods and Quantitation Limits
Legend Technical Services, Soils
UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Metals																	
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD Recovery			MS / MSD %RPD	LCS / LCSD Recovery			LCS / LCSD %RPD	MN Tier 1 Residential SRV	MN Tier 2 Industrial SRV	MN Tier 2 Recreation SRV	Unit
Antimony	6020A	7440-36-0	4.70	100	ug/kg	75	-	125	20	80	-	120	20	12,000	100,000	16,000	ug/kg
Arsenic	6020A	7440-38-2	18	250	ug/kg	75	-	125	20	80	-	120	20	9,000	20,000	11,000	ug/kg
Barium	6020A	7440-39-3	2.70	100	ug/kg	75	-	125	20	80	-	120	20	1,100,000	18,000,000	1,100,000	ug/kg
Boron	6020A	7440-42-8	260	2500	ug/kg	75	-	125	20	80	-	120	20	6,000,000	47,000,000	8,000,000	ug/kg
Cadmium	6020A	7440-43-9	9.70	100	ug/kg	75	-	125	20	80	-	120	20	25,000	200,000	35,000	ug/kg
Chromium	6020A	7440-47-3	16	250	ug/kg	75	-	125	20	80	-	120	20	87,000	650,000	120,000	ug/kg
Lead	6020A	7439-92-1	19	100	ug/kg	75	-	125	20	80	-	120	20	300,000	700,000	300,000	ug/kg
Selenium	6020A	7782-49-2	200	1000	ug/kg	75	-	125	20	80	-	120	20	160,000	1,300,000	200,000	ug/kg
Silver	6020A	7440-22-4	11	100	ug/kg	75	-	125	20	80	-	120	20	160,000	1,300,000	200,000	ug/kg
Thallium	6020A	7440-28-0	5.40	100	ug/kg	75	-	125	20	80	-	120	20	3,000	21,000	3,000	ug/kg
Mercury	7473	7439-97-6	1	50	ug/kg	80	-	120	20	80	-	120	20	500	1,500	1,200	ug/kg
Antimony	6010C	7440-36-0	190	1000	ug/kg	75	-	125	20	80	-	120	20	12,000	100,000	16,000	ug/kg
Arsenic	6010C	7440-38-2	290	1000	ug/kg	75	-	125	20	80	-	120	20	9,000	20,000	11,000	ug/kg
Barium	6010C	7440-39-3	100	1000	ug/kg	75	-	125	20	80	-	120	20	1,100,000	18,000,000	1,100,000	ug/kg
Boron	6010C	7440-42-8	540	2500	ug/kg	75	-	125	20	80	-	120	20	6,000,000	47,000,000	8,000,000	ug/kg
Cadmium	6010C	7440-43-9	10	50	ug/kg	75	-	125	20	80	-	120	20	25,000	200,000	35,000	ug/kg
Chromium	6010C	7440-47-3	20	500	ug/kg	75	-	125	20	80	-	120	20	87,000	650,000	120,000	ug/kg
Lead	6010C	7439-92-1	180	750	ug/kg	75	-	125	20	80	-	120	20	300,000	700,000	300,000	ug/kg
Selenium	6010C	7782-49-2	660	2500	ug/kg	75	-	125	20	80	-	120	20	160,000	1,300,000	200,000	ug/kg
Silver	6010C	7440-22-4	150	500	ug/kg	75	-	125	20	80	-	120	20	160,000	1,300,000	200,000	ug/kg
Thallium	6010C	7440-28-0	410	2000	ug/kg	75	-	125	20	80	-	120	20	3,000	21,000	3,000	ug/kg
PAHs																	
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD [2] Recovery			MS / MSD %RPD	LCS / LCSD [2] Recovery			LCS / LCSD %RPD	MN Tier 1 Residential SRV	MN Tier 2 Industrial SRV	MN Tier 2 Recreation SRV	Unit
2-Chloronaphthalene[3]	8270D	91-58-7	0.068	0.33	mg/kg	50	-	150	20	50	-	150	20				
2-Methylnaphthalene[3]	8270D	91-57-6	0.08	0.33	mg/kg	50	-	150	20	50	-	150	20	100	369	120	mg/kg
Acenaphthene[3]	8270D	83-32-9	0.063	0.33	mg/kg	50	-	150	20	50	-	150	20	1200	5260	1860	mg/kg
Acenaphthylene	8270D	208-96-8	0.071	0.33	mg/kg	40	-	100	20	55	-	95	20				
Anthracene	8270D	120-12-7	0.069	0.33	mg/kg	45	-	100	20	60	-	100	20	7880	45400	10000	mg/kg
Benzo(a)anthracene	8270D	56-55-3	0.065	0.33	mg/kg	45	-	100	20	55	-	100	20				
Benzo(a)pyrene	8270D	50-32-8	0.07	0.33	mg/kg	40	-	100	20	55	-	100	20				
Benzo(b)fluoranthene	8270D	205-99-2	0.059	0.33	mg/kg	40	-	100	20	55	-	100	20				
Benzo(g,h,i)perylene	8270D	191-24-2	0.071	0.33	mg/kg	35	-	110	20	50	-	100	20				
Benzo(k)fluoranthene	8270D	207-08-9	0.07	0.33	mg/kg	40	-	100	20	55	-	100	20				
Chrysene	8270D	218-01-9	0.064	0.33	mg/kg	40	-	100	20	50	-	100	20				
Dibenz(a,h)anthracene	8270D	53-70-3	0.082	0.33	mg/kg	35	-	110	20	50	-	100	20				
Fluoranthene	8270D	206-44-0	0.068	0.33	mg/kg	45	-	100	20	55	-	100	20	1080	6800	1290	mg/kg
Fluorene	8270D	86-73-7	0.065	0.33	mg/kg	45	-	100	20	55	-	95	20	850	4120	1200	mg/kg
Indeno (1,2,3-cd) pyrene	8270D	193-39-5	0.072	0.33	mg/kg	35	-	110	20	55	-	110	20				
Naphthalene	8270D	91-20-3	0.071	0.33	mg/kg	35	-	100	20	50	-	95	20	10	28	24	mg/kg
Phenanthrene	8270D	85-01-8	0.066	0.33	mg/kg	45	-	100	20	60	-	100	20				
Pyrene[3]	8270D	129-00-0	0.059	0.33	mg/kg	50	-	150	20	50	-	150	20	890	5800	1060	mg/kg
PAH Extended List																	-
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD [2] Recovery			MS / MSD %RPD	LCS / LCSD [2] Recovery			LCS / LCSD %RPD	MN Tier 1 Residential SRV	MN Tier 2 Industrial SRV	MN Tier 2 Recreation SRV	Unit
1,6-Dinitropyrene[3]	8270D	42397-64-8	0.045	0.55	mg/kg	50	-	150	40	50	-	150	40				
1,8-Dinitropyrene[3]	8270D	42397-65-9	0.024	0.28	mg/kg	50	-	150	40	50	-	150	40				
1-Nitropyrene[3]	8270D	5522-43-0	0.011	0.11	mg/kg	50	-	150	40	50	-	150	40				

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Table 1a
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Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MN Tier 1 Residential SRV	MN Tier 2 Industrial SRV	MN Tier 2 Recreation SRV	Unit
						Recovery			%RPD	Recovery			%RPD				
2-Methylnaphthalene[3]	8270D	91-57-6	0.031	0.11	mg/kg	50	-	150	40	50	-	150	40	100	369	120	mg/kg
2-Nitrofluorene[3]	8270D	607-57-8	0.016	0.11	mg/kg	50	-	150	40	50	-	150	40				
3-Methylcholanthrene[3]	8270D	56-49-5	0.01	0.11	mg/kg	50	-	150	40	50	-	150	40				
4-Nitropyrene[3]	8270D	57835-92-4	0.016	0.13	mg/kg	50	-	150	40	50	-	150	40				
5-Methylchrysene	8270D	3697-24-3	0.012	0.11	mg/kg	43.1	-	112	40	50.7	-	117	40				
5-Nitroacenaphthene[3]	8270D	602-87-9	0.014	0.11	mg/kg	50	-	150	40	50	-	150	40				
6-Nitrochrysene[3]	8270D	7496-02-8	0.013	0.22	mg/kg	50	-	150	40	50	-	150	40				
7,12-Dimethylbenz (a) anthracene	8270D	57-97-6	0.015	0.11	mg/kg	42.6	-	99.9	40	38.6	-	121	40				
7H-Dibenzo(c,g)carbazole[3]	8270D	194-59-2	0.015	0.055	mg/kg	50	-	150	40	50	-	150	40				
Acenaphthene	8270D	83-32-9	0.028	0.11	mg/kg	48.3	-	91	39	34.4	-	107	40	1200	5260	1860	mg/kg
Acenaphthylene	8270D	208-96-8	0.023	0.11	mg/kg	41.8	-	94.2	38.2	33.3	-	104	40				
Anthracene	8270D	120-12-7	0.016	0.11	mg/kg	54	-	98.9	40	54.8	-	105	40	7880	45400	10000	mg/kg
Benzo(a)anthracene	8270D	56-55-3	0.016	0.11	mg/kg	50.1	-	115	40	56.6	-	108	40				
Benzo(a)pyrene	8270D	50-32-8	0.0087	0.11	mg/kg	35.5	-	97.3	40	38	-	114	40				
Benzo(b&j)fluoranthene	8270D	205-99-2/205-82-3	0.02	0.22	mg/kg	30	-	121	40	38.4	-	122	40				
Benzo(e)pyrene[3]	8270D	192-97-2	0.013	0.11	mg/kg	50	-	150	40	50	-	150	40				
Benzo(g,h,i)perylene	8270D	191-24-2	0.013	0.11	mg/kg	33.2	-	96.4	40	50.9	-	101	40				
Benzo(k)fluoranthene	8270D	207-08-9	0.019	0.11	mg/kg	37.3	-	90.2	40	38	-	114	40				
Carbazole	8270D	86-74-8	0.014	0.11	mg/kg	49.1	-	105	40	54.1	-	104	40	700	1310	720	mg/kg
Chrysene	8270D	218-01-9	0.013	0.11	mg/kg	52.1	-	113	40	56.5	-	110	40				
Dibenz [a,h] acridine[3]	8270D	226-36-8	0.0046	0.11	mg/kg	50	-	150	40	50	-	150	40				
Dibenz(a,h)anthracene	8270D	53-70-3	0.011	0.11	mg/kg	30	-	97	40	47.9	-	100	40				
Dibenz(a,j)acridine[3]	8270D	224-42-0	0.0091	0.11	mg/kg	50	-	150	40	50	-	150	40				
Dibenzo(a,e)pyrene	8270D	192-65-4	0.01	0.11	mg/kg	30	-	103	40	30	-	108	40				
Dibenzo(a,h)pyrene	8270D	189-64-0	0.015	0.11	mg/kg	30	-	105	40	48.8	-	106	40				
Dibenzo(a,i)pyrene	8270D	189-55-9	0.012	0.11	mg/kg	30	-	108	40	45.7	-	98.8	40				
Dibenzo(a,l)pyrene	8270D	191-30-0	0.017	0.11	mg/kg	30	-	109	40	49.4	-	106	40				
Fluoranthene	8270D	206-44-0	0.013	0.11	mg/kg	39.3	-	123	40	54.9	-	108	40	1080	6800	1290	mg/kg
Fluorene	8270D	86-73-7	0.023	0.11	mg/kg	51.1	-	95.7	38	40.2	-	106	40	850	4120	1200	mg/kg
Indeno (1,2,3-cd) pyrene	8270D	193-39-5	0.013	0.11	mg/kg	35.6	-	90.4	40	36.2	-	112	40				
Naphthalene	8270D	91-20-3	0.032	0.11	mg/kg	40.4	-	96.2	30.8	30	-	114	40	10	28	24	mg/kg
Perylene[3]	8270D	198-55-0	0.013	0.11	mg/kg	50	-	150	40	50	-	150	40				
Phenanthrene	8270D	85-01-8	0.015	0.11	mg/kg	35.7	-	124	40	51.9	-	104	40				
Pyrene	8270D	129-00-0	0.016	0.11	mg/kg	32.7	-	140	40	58.3	-	108	40	890	5800	1060	mg/kg
Quinoline[3]	8270D	91-22-5	0.03	0.11	mg/kg	50	-	150	40	50	-	150	40	4	7	4	mg/kg
PCBs																	
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD [(2)]				LCS / LCSD [(2)]				MN Tier 1 Residential SRV	MN Tier 2 Ind SRV	MN Tier 2 Recreation SRV	
						Recovery			%RPD	Recovery			%RPD				
Aroclor 1016[3]	8082A	12674-11-2	0.01	0.2	mg/kg	70	-	130	20	70	-	130	20				
Aroclor 1221	8082A	11104-28-2	0.017	0.2	mg/kg												
Aroclor 1232	8082A	11141-16-5	0.013	0.2	mg/kg												
Aroclor 1242	8082A	53469-21-9	0.018	0.2	mg/kg												
Aroclor 1248	8082A	12672-29-6	0.011	0.2	mg/kg												
Aroclor 1254	8082A	11097-69-1	0.012	0.2	mg/kg												
Aroclor 1260	8082A	11096-82-5	0.0097	0.2	mg/kg	70	-	130	17.2	70	-	130	20				
SVOCs																	
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD [(2)]				LCS / LCSD [(2)]				MN Tier 1 Residential SRV	MN Tier 2 Ind SRV	MN Tier 2 Recreation SRV	Unit
						Recovery			%RPD	Recovery			%RPD				
1,2,4-Trichlorobenzene	8270D	120-82-1	0.075	0.33	mg/kg	35	-	100	20	50	-	100	20	200	985	290	mg/kg
1,2-Dichlorobenzene[3]	8270D	95-50-1	0.067	0.33	mg/kg	50	-	150	20	50	-	150	20	26	75	63	mg/kg

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Table 1a
Analytical Parameters, Methods and Quantitation Limits
Legend Technical Services, Soils
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Dakota County, Minnesota

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MN Tier 1 Residential SRV	MN Tier 2 Industrial SRV	MN Tier 2 Recreation SRV	Unit
						Recovery			%RPD	Recovery			%RPD				
1,2-Diphenylhydrazine as Azobenzene[3]	8270D	103-33-3	0.058	0.33	mg/kg	50	-	150	20	50	-	150	20				
1,3-Dichlorobenzene[3]	8270D	541-73-1	0.069	0.33	mg/kg	50	-	150	20	50	-	150	20	26	200	32	mg/kg
1,4-Dichlorobenzene	8270D	106-46-7	0.068	0.33	mg/kg	30	-	85	20	40	-	80	20	30	50	72	mg/kg
2,3,4,6-Tetrachlorophenol[3]	8270D	58-90-2	0.19	0.67	mg/kg	50	-	150	20	50	-	150	20	636	3700	700	mg/kg
2,4,5-Trichlorophenol[3]	8270D	95-95-4	0.071	0.67	mg/kg	50	-	150	20	50	-	150	20	1920	10600	2212	mg/kg
2,4,6-Trichlorophenol[3]	8270D	88-06-2	0.16	0.67	mg/kg	50	-	150	20	50	-	150	20	595	1060	705	mg/kg
2,4-Dichlorophenol[3]	8270D	120-83-2	0.15	0.67	mg/kg	50	-	150	20	50	-	150	20	48	230	61	mg/kg
2,4-Dimethylphenol[3]	8270D	105-67-9	0.13	0.67	mg/kg	50	-	150	20	50	-	150	20	390	1925	530	mg/kg
2,4-Dinitrophenol[3]	8270D	51-28-5	0.071	0.67	mg/kg	50	-	150	20	50	-	150	20				
2,4-Dinitrotoluene	8270D	121-14-2	0.068	0.33	mg/kg	45	-	95	20	50	-	90	20	50	355	60	mg/kg
2,6-Dichlorophenol[3]	8270D	87-65-0	0.13	0.67	mg/kg	50	-	150	20	50	-	150	20				
2,6-Dinitrotoluene[3]	8270D	606-20-2	0.077	0.33	mg/kg	50	-	150	20	50	-	150	20	25	175	30	mg/kg
2-Chloronaphthalene[3]	8270D	91-58-7	0.068	0.33	mg/kg	50	-	150	20	50	-	150	20				
2-Chlorophenol	8270D	95-57-8	0.15	0.67	mg/kg	35	-	100	20	50	-	85	20				
2-Methylnaphthalene[3]	8270D	91-57-6	0.08	0.33	mg/kg	50	-	150	20	50	-	150	20	100	369	120	mg/kg
2-Methylphenol[3]	8270D	95-48-7	0.082	0.67	mg/kg	50	-	150	20	50	-	150	20	75	352	95	mg/kg
2-Nitroaniline[3]	8270D	88-74-4	0.069	0.33	mg/kg	50	-	150	20	50	-	150	20				
2-Nitrophenol[3]	8270D	88-75-5	0.18	0.67	mg/kg	50	-	150	20	50	-	150	20				
3&4-Methylphenol[3]	8270D	108-39-4/106-44-5	0.082	0.67	mg/kg	50	-	150	20	50	-	150	20				
3,3´-Dichlorobenzidine[3]	8270D	91-94-1	0.45	1.6	mg/kg	50	-	150	20	50	-	150	20	25	50	30	mg/kg
3-Nitroaniline[3]	8270D	99-09-2	0.072	0.33	mg/kg	50	-	150	20	50	-	150	20				
4,6-Dinitro-2-methylphenol[3]	8270D	534-52-1	0.11	0.67	mg/kg	50	-	150	20	50	-	150	20				
4-Bromophenyl phenyl ether[3]	8270D	101-55-3	0.068	0.33	mg/kg	50	-	150	20	50	-	150	20				
4-Chloro-3-methylphenol	8270D	59-50-7	0.14	0.67	mg/kg	35	-	100	20	55	-	90	20				
4-Chloroaniline[3]	8270D	106-47-8	0.067	0.67	mg/kg	50	-	150	20	50	-	150	20				
4-Chlorophenyl phenyl ether[3]	8270D	7005-72-3	0.068	0.33	mg/kg	50	-	150	20	50	-	150	20				
4-Nitroaniline[3]	8270D	100-01-6	0.083	0.33	mg/kg	50	-	150	20	50	-	150	20				
4-Nitrophenol	8270D	100-02-7	0.17	0.67	mg/kg	40	-	100	20	45	-	100	20				
Acenaphthene[3]	8270D	83-32-9	0.063	0.33	mg/kg	50	-	150	20	50	-	150	20	1200	5260	1860	mg/kg
Acenaphthylene[3]	8270D	208-96-8	0.071	0.33	mg/kg	50	-	150	20	50	-	150	20				
Aniline[3]	8270D	62-53-3	0.066	0.67	mg/kg	50	-	150	20	50	-	150	20				
Anthracene	8270D	120-12-7	0.069	0.33	mg/kg	55	-	100	20	55	-	95	20	7880	45400	10000	mg/kg
Benzidine[3]	8270D	92-87-5	0.44	2.5	mg/kg	50	-	150	20	50	-	150	20				
Benzo(a)anthracene	8270D	56-55-3	0.065	0.33	mg/kg	50	-	100	20	55	-	100	20				
Benzo(a)pyrene	8270D	50-32-8	0.07	0.33	mg/kg	50	-	100	20	55	-	100	20				
Benzo(b)fluoranthene[3]	8270D	205-99-2	0.059	0.33	mg/kg	50	-	150	20	50	-	150	20				
Benzo(g,h,i)perylene[3]	8270D	191-24-2	0.071	0.33	mg/kg	50	-	150	20	50	-	150	20				
Benzo(k)fluoranthene[3]	8270D	207-08-9	0.07	0.33	mg/kg	50	-	150	20	50	-	150	20				
Benzoic acid[3]	8270D	65-85-0	0.064	0.33	mg/kg	50	-	150	20	50	-	150	20	50000	100000	83000	mg/kg
Benzyl alcohol[3]	8270D	100-51-6	0.15	0.67	mg/kg	50	-	150	20	50	-	150	20	8700	56000	9500	mg/kg
Bis(2-chloroethoxy)methane[3]	8270D	111-91-1	0.077	0.33	mg/kg	50	-	150	20	50	-	150	20				
Bis(2-chloroethyl)ether[3]	8270D	111-44-4	0.069	0.33	mg/kg	50	-	150	20	50	-	150	20	2.5	5	6	mg/kg
Bis(2-chloroisopropyl)ether[3]	8270D	108-60-1	0.078	0.33	mg/kg	50	-	150	20	50	-	150	20				
Bis(2-ethylhexyl)phthalate[3]	8270D	117-81-7	0.081	0.33	mg/kg	50	-	150	20	50	-	150	20	570	2100	690	mg/kg
Butyl benzyl phthalate[3]	8270D	85-68-7	0.083	0.33	mg/kg	50	-	150	20	50	-	150	20	580	3700	623	mg/kg
Carbazole[3]	8270D	86-74-8	0.076	0.33	mg/kg	50	-	150	20	50	-	150	20	700	1310	720	mg/kg
Chrysene	8270D	218-01-9	0.064	0.33	mg/kg	50	-	100	20	55	-	100	20				
Dibenz(a,h)anthracene[3]	8270D	53-70-3	0.082	0.33	mg/kg	50	-	150	20	50	-	150	20				
Dibenzofuran[3]	8270D	132-64-9	0.068	0.33	mg/kg	50	-	150	20	50	-	150	20	104	810	130	mg/kg
Diethyl phthalate[3]	8270D	84-66-2	0.063	0.33	mg/kg	50	-	150	20	50	-	150	20				

UMP023005

Table 1a
Analytical Parameters, Methods and Quantitation Limits
Legend Technical Services, Soils
UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD Recovery			MS / MSD %RPD	LCS / LCSD Recovery			LCS / LCSD %RPD	MN Tier 1 Residential SRV	MN Tier 2 Industrial SRV	MN Tier 2 Recreation SRV	Unit
Dimethyl phthalate[3]	8270D	131-11-3	0.069	0.33	mg/kg	50	-	150	20	50	-	150	20				
Di-n-butyl phthalate[3]	8270D	84-74-2	0.079	0.33	mg/kg	50	-	150	20	50	-	150	20	2440	16300	3070	mg/kg
Di-n-octyl phthalate[3]	8270D	117-84-0	0.1	0.33	mg/kg	50	-	150	20	50	-	150	20	520	3700	630	mg/kg
Fluoranthene	8270D	206-44-0	0.068	0.33	mg/kg	50	-	100	20	55	-	95	20	1080	6800	1290	mg/kg
Fluorene	8270D	86-73-7	0.065	0.33	mg/kg	50	-	100	20	55	-	95	20	850	4120	1200	mg/kg
Hexachlorobenzene[3]	8270D	118-74-1	0.062	0.33	mg/kg	50	-	150	20	50	-	150	20	5	9	8	mg/kg
Hexachlorobutadiene[3]	8270D	87-68-3	0.077	0.33	mg/kg	50	-	150	20	50	-	150	20	6	37	6	mg/kg
Hexachlorocyclopentadiene[3]	8270D	77-47-4	0.069	0.33	mg/kg	50	-	150	20	50	-	150	20	2	6	5	mg/kg
Hexachloroethane[3]	8270D	67-72-1	0.082	0.33	mg/kg	50	-	150	20	50	-	150	20				
Indeno (1,2,3-cd) pyrene[3]	8270D	193-39-5	0.072	0.33	mg/kg	50	-	150	20	50	-	150	20				
Isophorone[3]	8270D	78-59-1	0.076	0.33	mg/kg	50	-	150	20	50	-	150	20				
Naphthalene[3]	8270D	91-20-3	0.071	0.33	mg/kg	50	-	150	20	50	-	150	20	10	28	24	mg/kg
Nitrobenzene[3]	8270D	98-95-3	0.08	0.33	mg/kg	50	-	150	20	50	-	150	20				
N-Nitrosodimethylamine[3]	8270D	62-75-9	0.07	0.33	mg/kg	50	-	150	20	50	-	150	20				
N-Nitrosodi-n-propylamine	8270D	621-64-7	0.073	0.33	mg/kg	35	-	100	20	50	-	90	20	0.7	1.2	1.2	mg/kg
N-Nitrosodiphenylamine[3]	8270D	86-30-6	0.067	0.33	mg/kg	50	-	150	20	50	-	150	20	1950	3720	2585	mg/kg
Pentachlorophenol	8270D	87-86-5	0.19	0.67	mg/kg	30	-	100	20	35	-	95	20	80	120	80	mg/kg
Phenanthrene	8270D	85-01-8	0.066	0.33	mg/kg	55	-	100	20	55	-	95	20				
Phenol	8270D	108-95-2	0.14	0.67	mg/kg	35	-	100	20	50	-	85	20	1500	20203	1500	mg/kg
Pyrene[3]	8270D	129-00-0	0.059	0.33	mg/kg	50	-	150	20	50	-	150	20	890	5800	1060	mg/kg
VOCs																	
Parameter	**EPA Method**	**CAS#**	**MDL**	**RL**	**Unit**	**MS / MSD [2] Recovery**			**MS / MSD [2] %RPD**	**LCS / LCSD [2] Recovery**			**LCS / LCSD [2] %RPD**	**MN Tier 1 Residential SRV**	**MN Tier 2 Industrial SRV**	**MN Tier 2 Recreation SRV**	**Unit**
1,1,1,2-Tetrachloroethane[3]	8260B	630-20-6	0.02	0.2	mg/kg	70	-	130	20	70	-	130	20	31	51	83	mg/kg
1,1,1-Trichloroethane[3]	8260B	71-55-6	0.023	0.2	mg/kg	70	-	130	20	70	-	130	20	140	472	280	mg/kg
1,1,2,2-Tetrachloroethane	8260B	79-34-5	0.016	0.2	mg/kg	75	-	125	20	75	-	120	20	3.5	6.5	4	mg/kg
1,1,2-Trichloroethane[3]	8260B	79-00-5	0.014	0.2	mg/kg	70	-	130	20	70	-	130	20	9	14	24	mg/kg
1,1,2-Trichlorotrifluoroethane[3]	8260B	76-13-1	0.02	0.2	mg/kg	70	-	130	20	70	-	130	20	3745	5430	5430	mg/kg
1,1-Dichloroethane	8260B	75-34-3	0.0097	0.2	mg/kg	78.7	-	123	20	79.6	-	120	20	34	55	97	mg/kg
1,1-Dichloroethene	8260B	75-35-4	0.013	0.2	mg/kg	75.8	-	121	20	78.3	-	120	20	20	60	50	mg/kg
1,1-Dichloropropene[3]	8260B	563-58-6	0.015	0.2	mg/kg	80	-	120	20	80	-	120	20				
1,2,3-Trichlorobenzene[3]	8260B	87-61-6	0.097	0.5	mg/kg	80	-	120	20	80	-	120	20				
1,2,3-Trichloropropane[3]	8260B	96-18-4	0.03	0.2	mg/kg	80	-	120	20	80	-	120	20				
1,2,4-Trichlorobenzene[3]	8260B	120-82-1	0.071	0.5	mg/kg	80	-	120	20	80	-	120	20	200	985	290	mg/kg
1,2,4-Trimethylbenzene[3]	8260B	95-63-6	0.018	0.2	mg/kg	80	-	120	20	80	-	120	20	8	25	20	mg/kg
1,2-Dibromo-3-chloropropane[3]	8260B	96-12-8	0.046	0.5	mg/kg	80	-	120	20	80	-	120	20				
1,2-Dibromoethane (EDB)[3]	8260B	106-93-4	0.024	0.2	mg/kg	80	-	120	20	80	-	120	20	0.3	0.5	1	mg/kg
1,2-Dichlorobenzene[3]	8260B	95-50-1	0.013	0.2	mg/kg	80	-	120	20	80	-	120	20	26	75	63	mg/kg
1,2-Dichloroethane[3]	8260B	107-06-2	0.022	0.2	mg/kg	80	-	120	20	80	-	120	20	4	6	10	mg/kg
1,2-Dichloropropane[3]	8260B	78-87-5	0.021	0.2	mg/kg	80	-	120	20	80	-	120	20	4	6	11	mg/kg
1,3,5-Trimethylbenzene	8260B	108-67-8	0.025	0.2	mg/kg	75	-	120	20	77	-	120	20	3	10	8	mg/kg
1,3-Dichlorobenzene[3]	8260B	541-73-1	0.009	0.2	mg/kg	80	-	120	20	80	-	120	20	26	200	32	mg/kg
1,3-Dichloropropane[3]	8260B	142-28-9	0.015	0.2	mg/kg	80	-	120	20	80	-	120	20				
1,4-Dichlorobenzene	8260B	106-46-7	0.016	0.2	mg/kg	75	-	125	20	75	-	125	20	30	50	72	mg/kg
2,2-Dichloropropane[3]	8260B	594-20-7	0.052	0.2	mg/kg	80	-	120	20	80	-	120	20				
2-Butanone[3]	8260B	78-93-9	0.094	1	mg/kg	80	-	120	20	80	-	120	20				
2-Chlorotoluene	8260B	95-49-8	0.02	0.2	mg/kg	75	-	120	20	75.9	-	120	20	436	436	436	mg/kg
4-Chlorotoluene[3]	8260B	106-43-4	0.022	0.2	mg/kg	80	-	120	20	80	-	120	20				
Acetone[3]	8260B	67-64-1	0.12	1	mg/kg	80	-	120	20	80	-	120	20	340	1000	850	mg/kg
Allyl chloride[3]	8260B	107-05-1	0.025	0.2	mg/kg	80	-	120	20	80	-	120	20				

UMP023006

Table 1a
Analytical Parameters, Methods and Quantitation Limits
Legend Technical Services, Soils
UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MN Tier 1 Residential SRV	MN Tier 2 Industrial SRV	MN Tier 2 Recreation SRV	Unit
						Recovery			%RPD	Recovery			%RPD				
Benzene	8260B	71-43-2	0.015	0.2	mg/kg	80	-	120	20	80	-	120	20	6	10	14	mg/kg
Bromobenzene[3]	8260B	108-86-1	0.02	0.2	mg/kg	80	-	120	20	80	-	120	20				
Bromochloromethane[3]	8260B	74-97-5	0.023	0.2	mg/kg	80	-	120	20	80	-	120	20				
Bromodichloromethane[3]	8260B	75-27-4	0.019	0.2	mg/kg	80	-	120	20	80	-	120	20	10	17	28	mg/kg
Bromoform	8260B	75-25-2	0.036	0.2	mg/kg	80	-	120	20	80	-	120	20	370	650	630	mg/kg
Bromomethane[3]	8260B	74-83-9	0.03	0.2	mg/kg	80	-	120	20	80	-	120	20	0.7	2	2	mg/kg
Carbon tetrachloride[3]	8260B	56-23-5	0.025	0.2	mg/kg	80	-	120	20	80	-	120	20	0.3	0.9	0.7	mg/kg
Chlorobenzene	8260B	108-90-7	0.014	0.2	mg/kg	80	-	120	20	80	-	120	20	11	32	23	mg/kg
Chloroethane[3]	8260B	75-00-3	0.03	0.2	mg/kg	80	-	120	20	80	-	120	20	1000	3000	2250	mg/kg
Chloroform	8260B	67-66-3	0.031	0.2	mg/kg	80	-	120	20	80	-	120	20	2.5	4	7	mg/kg
Chloromethane[3]	8260B	74-87-3	0.027	0.2	mg/kg	80	-	120	20	80	-	120	20	8	23	20	mg/kg
cis-1,2-Dichloroethene[3]	8260B	156-59-2	0.012	0.2	mg/kg	80	-	120	20	80	-	120	20	8	22	19	mg/kg
cis-1,3-Dichloropropene[3]	8260B	10061-01-5	0.025	0.2	mg/kg	80	-	120	20	80	-	120	20				
Dibromochloromethane[3]	8260B	124-48-1	0.025	0.2	mg/kg	80	-	120	20	80	-	120	20	12	20	30	mg/kg
Dibromomethane[3]	8260B	74-95-3	0.025	0.2	mg/kg	80	-	120	20	80	-	120	20	260	1860	316	mg/kg
Dichlorodifluoromethane[3]	8260B	75-71-8	0.037	0.2	mg/kg	80	-	120	20	80	-	120	20	16	50	42	mg/kg
Dichlorofluoromethane[3]	8260B	75-43-4	0.01	0.2	mg/kg	80	-	120	20	80	-	120	20				
Ethyl ether[3]	8260B	60-29-7	0.024	0.2	mg/kg	80	-	120	20	80	-	120	20				
Ethylbenzene	8260B	100-41-4	0.021	0.2	mg/kg	80	-	120	20	80	-	120	20	200	200	200	mg/kg
Hexachlorobutadiene[3]	8260B	87-68-3	0.079	0.5	mg/kg	80	-	120	20	80	-	120	20	6	37	6	mg/kg
Isopropylbenzene[3]	8260B	98-82-8	0.03	0.2	mg/kg	80	-	120	20	80	-	120	20	30	87	74	mg/kg
m,p-Xylene[3]	8260B	136777-61-2	0.048	0.4	mg/kg	80	-	120	20	80	-	120	20				
Methyl isobutyl ketone[3]	8260B	108-10-1	0.043	0.2	mg/kg	80	-	120	20	80	-	120	20	1700	9000	2500	mg/kg
Methyl tert-butyl ether[3]	8260B	1634-04-4	0.0097	0.2	mg/kg	80	-	120	20	80	-	120	20				
Methylene chloride[3]	8260B	75-09-2	0.06	0.5	mg/kg	80	-	120	20	80	-	120	20	97	158	270	mg/kg
Naphthalene[3]	8260B	91-20-3	0.048	0.5	mg/kg	80	-	120	20	80	-	120	20	10	28	24	mg/kg
n-Butylbenzene	8260B	104-51-8	0.016	0.2	mg/kg	73.8	-	125	20	75	-	125	20	30	92	70	mg/kg
n-Propylbenzene	8260B	103-65-1	0.01	0.2	mg/kg	75	-	120	20	75	-	120	20	30	93	70	mg/kg
o-Xylene[3]	8260B	95-47-6	0.017	0.2	mg/kg	80	-	120	20	80	-	120	20				
p-Isopropyltoluene[3]	8260B	99-87-6	0.011	0.2	mg/kg	80	-	120	20	80	-	120	20				
sec-Butylbenzene[3]	8260B	135-98-8	0.022	0.2	mg/kg	80	-	120	20	80	-	120	20	25	70	55	mg/kg
Styrene[3]	8260B	100-42-5	0.016	0.2	mg/kg	80	-	120	20	80	-	120	20	210	600	500	mg/kg
tert-Butylbenzene[3]	8260B	98-06-6	0.026	0.2	mg/kg	80	-	120	20	80	-	120	20	30	90	55	mg/kg
Tetrachloroethene[3]	8260B	127-18-4	0.038	0.2	mg/kg	80	-	120	20	80	-	120	20	72	131	145	mg/kg
Tetrahydrofuran[3]	8260B	109-99-9	0.11	1	mg/kg	80	-	120	20	80	-	120	20				
Toluene	8260B	108-88-3	0.0068	0.2	mg/kg	80	-	120	20	80	-	120	20	107	305	260	mg/kg
trans-1,2-Dichloroethene[3]	8260B	156-60-5	0.018	0.2	mg/kg	80	-	120	20	80	-	120	20	11	33	28	mg/kg
trans-1,3-Dichloropropene[3]	8260B	10061-02-6	0.02	0.2	mg/kg	80	-	120	20	80	-	120	20				
Trichloroethene	8260B	79-01-6	0.018	0.2	mg/kg	80	-	120	20	80	-	120	20	29	46	82	mg/kg
Trichlorofluoromethane[3]	8260B	75-69-4	0.029	0.2	mg/kg	80	-	120	20	80	-	120	20	67	195	168	mg/kg
Vinyl chloride	8260B	75-01-4	0.021	0.2	mg/kg	74.8	-	130	20	75	-	130	20	0.8	2.2	2	mg/kg

(1) The laboratory reporting limits (RL) and minimum detection limits (MDL) are periodically updated. The RL and MDL may vary based on level of moisture present (dry weight correction), initial volume and possible matrix interferences.

(2) The laboratory LCS/LCSD, MS/MSD spike recoveries and RPDs are periodically updated. The LCS/LCSD and MS/MSD spike recoveries and RPDs noted in the laboratory report will be used for data validation. Where the laboratory has no acceptance limits established, the interim values will be used during Barr data evaluation.

(3) Interim values to be used during Barr data evaluation until the laboratory establishes calculated spike recovery and RPD limits.

UMP023007

Table 1b
Analytical Parameters, Methods and Quantitation Limits
Legend Technical Services, Waters
UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Metals																
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
Antimony, Dissolved	6020A	7440-36-0	0.0031	0.2	ug/L	75	-	125	20	80	-	120	20	6	ug/L	HRL93
Arsenic, Dissolved	6020A	7440-38-2	0.1100	1	ug/L	75	-	125	20	80	-	120	20			
Barium, Dissolved	6020A	7440-39-3	0.012	0.2	ug/L	75	-	125	20	80	-	120	20	2000	ug/L	HRL93
Boron, Dissolved	6020A	7440-42-8	0.650	5.00	ug/L	75	-	125	20	80	-	120	20	1000	ug/L	RAA08
Cadmium, Dissolved	6020A	7440-43-9	0.0220	0.2	ug/L	75	-	125	20	80	-	120	20	0.5	ug/L	HRL15 (1)
Chromium, Dissolved	6020A	7440-47-3	0.0640	0.5	ug/L	75	-	125	20	80	-	120	20	100	ug/L	CR HRL93
Lead, Dissolved	6020A	7439-92-1	0.0370	0.2	ug/L	75	-	125	20	80	-	120	20			
Selenium, Dissolved	6020A	7782-49-2	0.170	1.0	ug/L	75	-	125	20	80	-	120	20	30	ug/L	HRL93
Silver, Dissolved	6020A	7440-22-4	0.0065	0.2	ug/L	75	-	125	20	80	-	120	20	30	ug/L	HRL93
Thallium, Dissolved	6020A	7440-28-0	0.0190	0.2	ug/L	75	-	125	20	80	-	120	20	0.6	ug/L	HRL94
Mercury, Dissolved & Total	6020A	7439-97-6	0.0190	0.200	ug/L	75	-	125	20	80	-	120	20			
Antimony	6010C	7440-36-0	3.9	20	ug/L	75	-	125	20	80	-	120	20	6	ug/L	HRL93
Arsenic	6010C	7440-38-2	5.8	20	ug/L	75	-	125	20	80	-	120	20			
Barium	6010C	7440-39-3	2.0	20	ug/L	75	-	125	20	80	-	120	20	2000	ug/L	HRL93
Boron	6010C	7440-42-8	11	50	ug/L	75	-	125	20	80	-	120	20	1000	ug/L	RAA08
Cadmium	6010C	7440-43-9	0.2	1.0	ug/L	75	-	125	20	80	-	120	20	0.5	ug/L	HRL15 (1)
Chromium	6010C	7440-47-3	0.40	10	ug/L	75	-	125	20	80	-	120	20	100	ug/L	CR HRL93
Lead	6010C	7439-92-1	3.6	15	ug/L	75	-	125	20	80	-	120	20			
Selenium	6010C	7782-49-2	13	50	ug/L	75	-	125	20	80	-	120	20	30	ug/L	HRL93
Silver	6010C	7440-22-4	3.0	10	ug/L	75	-	125	20	80	-	120	20	30	ug/L	HRL93
Thallium	6010C	7440-28-0	8.3	40	ug/L	75	-	125	20	80	-	120	20	0.6	ug/L	HRL94
PAHs																
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
2-Chloronaphthalene[3]	8270D	91-58-7	0.38	10	ug/L	50	-	150	20	50	-	150	20			
2-Methylnaphthalene[3]	8270D	91-57-6	0.7	10	ug/L	50	-	150	20	50	-	150	20	8	ug/L	RAA13
Acenaphthene[3]	8270D	83-32-9	0.41	10	ug/L	50	-	150	20	50	-	150	20	100	ug/L	HBV15
Acenaphthylene	8270D	208-96-8	0.38	10	ug/L	30	-	118	37.6	50	-	110	20			
Anthracene	8270D	120-12-7	0.36	10	ug/L	30	-	123	25.1	55	-	110	20	2000	ug/L	HRL93
Benzo(a)anthracene	8270D	56-55-3	0.23	10	ug/L	30	-	123	49.7	35	-	100	20			
Benzo(a)pyrene	8270D	50-32-8	0.34	10	ug/L	30	-	127	46.5	30	-	100	20	0.06	ug/L	HBV12 (1)
Benzo(b)fluoranthene	8270D	205-99-2	0.18	10	ug/L	30	-	118	48.9	30	-	100	20			
Benzo(g,h,i)perylene	8270D	191-24-2	0.43	10	ug/L	30	-	123	58.6	30	-	90	20			
Benzo(k)fluoranthene	8270D	207-08-9	0.55	10	ug/L	30	-	133	52.8	30	-	100	20			
Chrysene	8270D	218-01-9	0.34	10	ug/L	30	-	121	50.1	30	-	100	20			
Dibenz(a,h)anthracene	8270D	53-70-3	0.31	10	ug/L	30	-	123	56.6	30	-	90	20			
Fluoranthene	8270D	206-44-0	0.36	10	ug/L	30	-	125	53.1	55	-	110	20	70	ug/L	HBV15 (1)
Fluorene	8270D	86-73-7	0.35	10	ug/L	30	-	123	45.9	55	-	110	20	300	ug/L	HRL93
Indeno (1,2,3-cd) pyrene	8270D	193-39-5	0.37	10	ug/L	30	-	122	58.4	30	-	100	20			
Naphthalene	8270D	91-20-3	0.33	10	ug/L	30	-	132	30.8	45	-	110	20	70	ug/L	HRL13
Phenanthrene	8270D	85-01-8	0.28	10	ug/L	30.3	-	118	49.4	55	-	110	20			
Pyrene[3]	8270D	129-00-0	0.49	10	ug/L	50	-	150	20	50	-	150	20	50	ug/L	HBV15 (1)
PCBs																
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
Aroclor 1016[3]	8082A	12674-11-2	0.12	2	ug/L	70	-	130	20	70	-	130	20			
Aroclor 1221	8082A	11104-28-2	0.41	2	ug/L											
Aroclor 1232	8082A	11141-16-5	0.21	2	ug/L											

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Table 1b
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Legend Technical Services, Waters
UMore Park/Former Gopher Ordnance Works Remedial Investigation
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Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
Aroclor 1242	8082A	53469-21-9	0.13	2	ug/L											
Aroclor 1248	8082A	12672-29-6	0.15	2	ug/L											
Aroclor 1254	8082A	11097-69-1	0.13	2	ug/L											
Aroclor 1260	8082A	11096-82-5	0.1	2	ug/L	70	-	130	15.9	70	-	130	20			
SVOCs																
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
1,2,4-Trichlorobenzene	8270D	120-82-1	0.53	10	ug/L	30	-	100	20	38	-	100	20	4	ug/L	HRL13 (1)
1,2-Dichlorobenzene[3]	8270D	95-50-1	0.47	10	ug/L	50	-	150	20	50	-	150	20	600	ug/L	HRL93
1,2-Diphenylhydrazine as Azobenzene[3]	8270D	103-33-3	0.32	10	ug/L	50	-	150	20	50	-	150	20			
1,3-Dichlorobenzene[3]	8270D	541-73-1	0.43	10	ug/L	50	-	150	20	50	-	150	20			
1,4-Dichlorobenzene	8270D	106-46-7	0.32	10	ug/L	30	-	90	20	30	-	90	20	10	ug/L	HRL94
2,3,4,6-Tetrachlorophenol[3]	8270D	58-90-2	0.74	10	ug/L	50	-	150	20	50	-	150	20			
2,4,5-Trichlorophenol[3]	8270D	95-95-4	1.1	10	ug/L	50	-	150	20	50	-	150	20			
2,4,6-Trichlorophenol[3]	8270D	88-06-2	0.82	10	ug/L	50	-	150	20	50	-	150	20	30	ug/L	HRL93
2,4-Dichlorophenol[3]	8270D	120-83-2	0.78	10	ug/L	50	-	150	20	50	-	150	20	20	ug/L	HRL93
2,4-Dimethylphenol[3]	8270D	105-67-9	0.99	10	ug/L	50	-	150	20	50	-	150	20	100	ug/L	HRL93
2,4-Dinitrophenol[3]	8270D	51-28-5	0.7	10	ug/L	50	-	150	20	50	-	150	20	10	ug/L	HRL94
2,4-Dinitrotoluene	8270D	121-14-2	0.44	10	ug/L	30	-	110	20	57	-	100	20			
2,6-Dichlorophenol[3]	8270D	87-65-0	0.93	10	ug/L	50	-	150	20	50	-	150	20			
2,6-Dinitrotoluene[3]	8270D	606-20-2	0.39	10	ug/L	50	-	150	20	50	-	150	20			
2-Chloronaphthalene[3]	8270D	91-58-7	0.38	10	ug/L	50	-	150	20	50	-	150	20			
2-Chlorophenol	8270D	95-57-8	1.2	10	ug/L	30	-	100	20	45	-	95	20	30	ug/L	HRL93
2-Methylnaphthalene[3]	8270D	91-57-6	0.7	10	ug/L	50	-	150	20	50	-	150	20	8	ug/L	RAA13
2-Methylphenol[3]	8270D	95-48-7	1.4	10	ug/L	50	-	150	20	50	-	150	20	30	ug/L	HRL93
2-Nitroaniline[3]	8270D	88-74-4	0.83	10	ug/L	50	-	150	20	50	-	150	20			
2-Nitrophenol[3]	8270D	88-75-5	0.86	10	ug/L	50	-	150	20	50	-	150	20			
3&4-Methylphenol[3]	8270D	108-39-4/106-44-5	1.5	10	ug/L	50	-	150	20	50	-	150	20	3	ug/L	MP HRL94
3,3´-Dichlorobenzidine[3]	8270D	91-94-1	9.9	25	ug/L	50	-	150	20	50	-	150	20	0.8	ug/L	HRL93
3-Nitroaniline[3]	8270D	99-09-2	2	10	ug/L	50	-	150	20	50	-	150	20			
4,6-Dinitro-2-methylphenol[3]	8270D	534-52-1	1	10	ug/L	50	-	150	20	50	-	150	20			
4-Bromophenyl phenyl ether[3]	8270D	101-55-3	0.34	10	ug/L	50	-	150	20	50	-	150	20			
4-Chloro-3-methylphenol	8270D	59-50-7	0.68	10	ug/L	30	-	113	20	52	-	100	20			
4-Chloroaniline[3]	8270D	106-47-8	2.3	10	ug/L	50	-	150	20	50	-	150	20			
4-Chlorophenyl phenyl ether[3]	8270D	7005-72-3	0.45	10	ug/L	50	-	150	20	50	-	150	20			
4-Nitroaniline[3]	8270D	100-01-6	1.2	10	ug/L	50	-	150	20	50	-	150	20			
4-Nitrophenol	8270D	100-02-7	0.91	10	ug/L	30	-	112	34.3	30	-	100	20			
Acenaphthene[3]	8270D	83-32-9	0.41	10	ug/L	50	-	150	20	50	-	150	20	100	ug/L	HBV15
Acenaphthylene[3]	8270D	208-96-8	0.38	10	ug/L	50	-	150	20	50	-	150	20			
Aniline[3]	8270D	62-53-3	1.3	10	ug/L	50	-	150	20	50	-	150	20			
Anthracene	8270D	120-12-7	0.36	10	ug/L	30	-	119	20	60	-	100	20	2000	ug/L	HRL93
Benzidine[3]	8270D	92-87-5	8.2	100	ug/L	50	-	150	20	50	-	150	20			
Benzo(a)anthracene	8270D	56-55-3	0.23	10	ug/L	30	-	122	20	64	-	100	20			
Benzo(a)pyrene	8270D	50-32-8	0.34	10	ug/L	30	-	118	20	60	-	100	20	0.06	ug/L	HBV12 (1)
Benzo(b)fluoranthene[3]	8270D	205-99-2	0.18	10	ug/L	50	-	150	20	50	-	150	20			
Benzo(g,h,i)perylene[3]	8270D	191-24-2	0.43	10	ug/L	50	-	150	20	50	-	150	20			
Benzo(k)fluoranthene[3]	8270D	207-08-9	0.55	10	ug/L	50	-	150	20	50	-	150	20			
Benzoic acid[3]	8270D	65-85-0	1.8	10	ug/L	50	-	150	20	50	-	150	20	30000	ug/L	HRL93
Benzyl alcohol[3]	8270D	100-51-6	0.68	10	ug/L	50	-	150	20	50	-	150	20			

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Table 1b
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UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
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Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
Bis(2-chloroethoxy)methane[3]	8270D	111-91-1	0.41	10	ug/L	50	-	150	20	50	-	150	20			
Bis(2-chloroethyl)ether[3]	8270D	111-44-4	0.59	10	ug/L	50	-	150	20	50	-	150	20	0.3	ug/L	HRL93
Bis(2-chloroisopropyl)ether[3]	8270D	108-60-1	0.47	10	ug/L	50	-	150	20	50	-	150	20			
Bis(2-ethylhexyl)phthalate[3]	8270D	117-81-7	0.77	10	ug/L	50	-	150	20	50	-	150	20	7	ug/L	HRL15 (1)
Butyl benzyl phthalate[3]	8270D	85-68-7	0.67	10	ug/L	50	-	150	20	50	-	150	20	100	ug/L	HRL15
Carbazole[3]	8270D	86-74-8	0.42	10	ug/L	50	-	150	20	50	-	150	20			
Chrysene	8270D	218-01-9	0.34	10	ug/L	30	-	125	20	60	-	100	20			
Dibenz(a,h)anthracene[3]	8270D	53-70-3	0.31	10	ug/L	50	-	150	20	50	-	150	20			
Dibenzofuran[3]	8270D	132-64-9	0.77	10	ug/L	50	-	150	20	50	-	150	20			
Diethyl phthalate[3]	8270D	84-66-2	0.42	10	ug/L	50	-	150	20	50	-	150	20	6000	ug/L	HRL93
Dimethyl phthalate[3]	8270D	131-11-3	0.44	10	ug/L	50	-	150	20	50	-	150	20	70000	ug/L	HRL94
Di-n-butyl phthalate[3]	8270D	84-74-2	0.42	10	ug/L	50	-	150	20	50	-	150	20	20	ug/L	HRL15
Di-n-octyl phthalate[3]	8270D	117-84-0	0.48	10	ug/L	50	-	150	20	50	-	150	20			
Fluoranthene	8270D	206-44-0	0.36	10	ug/L	30	-	119	20	63	-	100	20	70	ug/L	HBV15 (1)
Fluorene	8270D	86-73-7	0.35	10	ug/L	30	-	107	20	59	-	100	20	300	ug/L	HRL93
Hexachlorobenzene[3]	8270D	118-74-1	0.3	10	ug/L	50	-	150	20	50	-	150	20	0.2	ug/L	HRL93
Hexachlorobutadiene[3]	8270D	87-68-3	0.37	10	ug/L	50	-	150	20	50	-	150	20	1	ug/L	HRL93
Hexachlorocyclopentadiene[3]	8270D	77-47-4	0.52	10	ug/L	50	-	150	20	50	-	150	20			
Hexachloroethane[3]	8270D	67-72-1	0.61	10	ug/L	50	-	150	20	50	-	150	20			
Indeno (1,2,3-cd) pyrene[3]	8270D	193-39-5	0.37	10	ug/L	50	-	150	20	50	-	150	20			
Isophorone[3]	8270D	78-59-1	0.45	10	ug/L	50	-	150	20	50	-	150	20	100	ug/L	HRL93
Naphthalene[3]	8270D	91-20-3	0.33	10	ug/L	50	-	150	20	50	-	150	20	70	ug/L	HRL13
Nitrobenzene[3]	8270D	98-95-3	0.51	10	ug/L	50	-	150	20	50	-	150	20			
N-Nitrosodimethylamine[3]	8270D	62-75-9	0.34	10	ug/L	50	-	150	20	50	-	150	20			
N-Nitrosodi-n-propylamine	8270D	621-64-7	0.47	10	ug/L	37	-	100	20	55	-	100	20			
N-Nitrosodiphenylamine[3]	8270D	86-30-6	0.54	10	ug/L	50	-	150	20	50	-	150	20	70	ug/L	HRL93
Pentachlorophenol	8270D	87-86-5	1.2	10	ug/L	30	-	130	20	45	-	107	20	0.3	ug/L	HRL15 (1)
Phenanthrene	8270D	85-01-8	0.28	10	ug/L	30	-	117	20	62	-	100	20			
Phenol	8270D	108-95-2	1.2	10	ug/L	30	-	80	31.9	30	-	80	20	4000	ug/L	HRL93
Pyrene[3]	8270D	129-00-0	0.49	10	ug/L	50	-	150	20	50	-	150	20	50	ug/L	HBV15 (1)
VOCs																
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
1,1,1,2-Tetrachloroethane[3]	8260B	630-20-6	0.024	1	ug/L	80	-	120	20	80	-	120	20	70	ug/L	HRL93
1,1,1-Trichloroethane[3]	8260B	71-55-6	0.069	1	ug/L	80	-	120	20	80	-	120	20	9000	ug/L	HRL09 (1)
1,1,2,2-Tetrachloroethane	8260B	79-34-5	0.051	1	ug/L	76.8	-	125	20	80	-	121	20	2	ug/L	HRL94
1,1,2-Trichloroethane[3]	8260B	79-00-5	0.1	1	ug/L	80	-	120	20	80	-	120	20	3	ug/L	HRL93
1,1,2-Trichlorotrifluoroethane[3]	8260B	76-13-1	0.081	1	ug/L	80	-	120	20	80	-	120	20	200000	ug/L	HRL93
1,1-Dichloroethane	8260B	75-34-3	0.05	1	ug/L	80	-	125	20	80	-	125	20	100	ug/L	RAA09 (1)
1,1-Dichloroethene	8260B	75-35-4	0.065	1	ug/L	80	-	125	20	80	-	125	20	200	ug/L	HRL11
1,1-Dichloropropene[3]	8260B	563-58-6	0.15	1	ug/L	80	-	120	20	80	-	120	20			
1,2,3-Trichlorobenzene[3]	8260B	87-61-6	0.45	5	ug/L	80	-	120	20	80	-	120	20			
1,2,3-Trichloropropane[3]	8260B	96-18-4	0.056	2.5	ug/L	80	-	120	20	80	-	120	20	0.003	ug/L	HRL13 (1)
1,2,4-Trichlorobenzene[3]	8260B	120-82-1	0.091	5	ug/L	80	-	120	20	80	-	120	20	4	ug/L	HRL13 (1)
1,2,4-Trimethylbenzene[3]	8260B	95-63-6	0.054	1	ug/L	80	-	120	20	80	-	120	20	100	ug/L	RAA10
1,2-Dibromo-3-chloropropane[3]	8260B	96-12-8	0.033	5	ug/L	80	-	120	20	80	-	120	20			
1,2-Dibromoethane (EDB)[3]	8260B	106-93-4	0.042	2.5	ug/L	80	-	120	20	80	-	120	20	0.004	ug/L	HRL93
1,2-Dichlorobenzene[3]	8260B	95-50-1	0.052	1	ug/L	80	-	120	20	80	-	120	20	600	ug/L	HRL93
1,2-Dichloroethane[3]	8260B	107-06-2	0.064	1	ug/L	80	-	120	20	80	-	120	20	1	ug/L	HRL13 (1)

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Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
1,2-Dichloropropane[3]	8260B	78-87-5	0.034	1	ug/L	80	-	120	20	80	-	120	20	5	ug/L	HRL94
1,3,5-Trimethylbenzene	8260B	108-67-8	0.046	1	ug/L	75	-	125	20	75.4	-	125	20	100	ug/L	HRL09
1,3-Dichlorobenzene[3]	8260B	541-73-1	0.068	1	ug/L	80	-	120	20	80	-	120	20			
1,3-Dichloropropane[3]	8260B	142-28-9	0.15	1	ug/L	80	-	120	20	80	-	120	20			
1,4-Dichlorobenzene	8260B	106-46-7	0.047	1	ug/L	75	-	125	20	75	-	125	20	10	ug/L	HRL94
2,2-Dichloropropane[3]	8260B	594-20-7	0.28	5	ug/L	80	-	120	20	80	-	120	20			
2-Butanone[3]	8260B	78-93-9	0.33	20	ug/L	80	-	120	20	80	-	120	20			
2-Chlorotoluene	8260B	95-49-8	0.052	1	ug/L	75	-	125	20	75.4	-	125	20			
4-Chlorotoluene[3]	8260B	106-43-4	0.041	1	ug/L	80	-	120	20	80	-	120	20			
Acetone[3]	8260B	67-64-1	0.32	20	ug/L	80	-	120	20	80	-	120	20	4000	ug/L	HRL11 (1)
Allyl chloride[3]	8260B	107-05-1	0.078	5	ug/L	80	-	120	20	80	-	120	20	30	ug/L	HRL94
Benzene	8260B	71-43-2	0.034	1	ug/L	80	-	120	20	80	-	120	20	2	ug/L	HRL09 (1)
Bromobenzene[3]	8260B	108-86-1	0.042	1	ug/L	80	-	120	20	80	-	120	20			
Bromochloromethane[3]	8260B	74-97-5	0.1	1	ug/L	80	-	120	20	80	-	120	20			
Bromodichloromethane[3]	8260B	75-27-4	0.042	1	ug/L	80	-	120	20	80	-	120	20	6	ug/L	HRL93
Bromoform	8260B	75-25-2	0.08	5	ug/L	80	-	120	20	80	-	120	20	40	ug/L	HRL93
Bromomethane[3]	8260B	74-83-9	0.17	5	ug/L	80	-	120	20	80	-	120	20	10	ug/L	HRL93
Carbon tetrachloride[3]	8260B	56-23-5	0.029	1	ug/L	80	-	120	20	80	-	120	20	1	ug/L	HRL13 (1)
Chlorobenzene	8260B	108-90-7	0.037	1	ug/L	80	-	120	20	80	-	120	20	100	ug/L	HRL93
Chloroethane[3]	8260B	75-00-3	0.062	2.5	ug/L	80	-	120	20	80	-	120	20	0	ug/L	ND RAA09
Chloroform	8260B	67-66-3	0.056	1	ug/L	79.8	-	125	20	80	-	123	20	30	ug/L	HRL09
Chloromethane[3]	8260B	74-87-3	0.062	2.5	ug/L	80	-	120	20	80	-	120	20			
cis-1,2-Dichloroethene[3]	8260B	156-59-2	0.097	1	ug/L	80	-	120	20	80	-	120	20	6	ug/L	HBV14 (1)
cis-1,3-Dichloropropene[3]	8260B	10061-01-5	0.041	1	ug/L	80	-	120	20	80	-	120	20	2	ug/L	DCP HRL94
Dibromochloromethane[3]	8260B	124-48-1	0.07	2.5	ug/L	80	-	120	20	80	-	120	20	10	ug/L	HRL93
Dibromomethane[3]	8260B	74-95-3	0.088	2.5	ug/L	80	-	120	20	80	-	120	20			
Dichlorodifluoromethane[3]	8260B	75-71-8	0.14	5	ug/L	80	-	120	20	80	-	120	20	700	ug/L	HRL11
Dichlorofluoromethane[3]	8260B	75-43-4	0.059	1	ug/L	80	-	120	20	80	-	120	20	30	ug/L	RAA15
Ethyl ether[3]	8260B	60-29-7	0.091	5	ug/L	80	-	120	20	80	-	120	20	200	ug/L	RAA10 (1)
Ethylbenzene	8260B	100-41-4	0.033	1	ug/L	80	-	120	20	80	-	120	20	50	ug/L	HRL11
Hexachlorobutadiene[3]	8260B	87-68-3	0.19	10	ug/L	80	-	120	20	80	-	120	20	1	ug/L	HRL93
Isopropylbenzene[3]	8260B	98-82-8	0.037	1	ug/L	80	-	120	20	80	-	120	20	300	ug/L	HRL93
m,p-Xylene[3]	8260B	136777-61-2	0.087	2	ug/L	80	-	120	20	80	-	120	20	300	ug/l	XYL HRL11 (1)
Methyl isobutyl ketone[3]	8260B	108-10-1	0.17	5	ug/L	80	-	120	20	80	-	120	20	300	ug/L	HRL94
Methyl tert-butyl ether[3]	8260B	1634-04-4	0.056	1	ug/L	80	-	120	20	80	-	120	20	60	ug/L	RAA13 (1)
Methylene chloride[3]	8260B	75-09-2	0.1	5	ug/L	80	-	120	20	80	-	120	20	5	ug/L	HRLMCL
Naphthalene[3]	8260B	91-20-3	0.032	5	ug/L	80	-	120	20	80	-	120	20	70	ug/L	HRL13
n-Butylbenzene	8260B	104-51-8	0.028	2.5	ug/L	75	-	130	20	75	-	125	20			
n-Propylbenzene	8260B	103-65-1	0.04	1	ug/L	75	-	125	20	75.8	-	125	20			
o-Xylene[3]	8260B	95-47-6	0.053	1	ug/L	80	-	120	20	80	-	120	20	300	ug/L	XYL HRL11 (1)
p-Isopropyltoluene[3]	8260B	99-87-6	0.052	2.5	ug/L	80	-	120	20	80	-	120	20			
sec-Butylbenzene[3]	8260B	135-98-8	0.055	1	ug/L	80	-	120	20	80	-	120	20			
Styrene[3]	8260B	100-42-5	0.048	1	ug/L	80	-	120	20	80	-	120	20			
tert-Butylbenzene[3]	8260B	98-06-6	0.028	1	ug/L	80	-	120	20	80	-	120	20			
Tetrachloroethene[3]	8260B	127-18-4	0.035	1	ug/L	80	-	120	20	80	-	120	20	4	ug/L	HBV14 (1)
Tetrahydrofuran[3]	8260B	109-99-9	0.34	20	ug/L	80	-	120	20	80	-	120	20			
Toluene	8260B	108-88-3	0.064	1	ug/L	80	-	120	20	80	-	120	20	200	ug/L	HRL11
trans-1,2-Dichloroethene[3]	8260B	156-60-5	0.058	1	ug/L	80	-	120	20	80	-	120	20	40	ug/L	HRL13 (1)
trans-1,3-Dichloropropene[3]	8260B	10061-02-6	0.067	1	ug/L	80	-	120	20	80	-	120	20	2	ug/L	DCP HRL94

Table 1b
Analytical Parameters, Methods and Quantitation Limits
Legend Technical Services, Waters
UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				MDH HUM Health		
						Recovery			%RPD	Recovery			%RPD	Limit	Unit	Basis
Trichloroethene	8260B	79-01-6	0.096	1	ug/L	80	-	120	20	80	-	120	20	0.4	ug/L	HRL15 (1)
Trichlorofluoromethane[3]	8260B	75-69-4	0.26	1	ug/L	80	-	120	20	80	-	120	20	2000	ug/L	HRL93
Vinyl chloride	8260B	75-01-4	0.046	1	ug/L	75	-	130	20	75	-	130	20	0.2	ug/L	HRL09 (1)

(1) The laboratory reporting limits (RL) and minimum detection limits (MDL) are periodically updated. The RL and MDL may vary based on level of moisture present (dry weight correction), initial volume and possible matrix interferences.
(2) The laboratory LCS/LCSD, MS/MSD spike recoveries and RPDs are periodically updated. The LCS/LCSD and MS/MSD spike recoveries and RPDs noted in the laboratory report will be used for data validation. Where the laboratory has no acceptance limits established, the interim values will be used during Barr data evaluation.
(3) Interim values, provided by MPCA, to be used during Barr data evaluation until the laboratory establishes calculated spike recovery and RPD limits.
MDH HUM Health = Minnesota Department of Health Human Health-Based Water Limits

UMP023012

Table 1e
Analytical Parameters, Methods and Quantitation Limits
ALS Semi Valley, Air
UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

TO-15														Assumes 6L Summa and may vary with canister pressure dilution factor and/or matrix effects.
Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD				
						Recovery			%RPD	Recovery				%RPD
Propene	TO-15	115-07-1	0.14	0.5	ug/m3					49	-	131		25
Dichlorodifluoromethane (CFC 12)	TO-15	75-71-8	0.17	0.5	ug/m3					65	-	117		25
Chloromethane	TO-15	74-87-3	0.15	0.5	ug/m3					48	-	132		25
1,2-Dichloro-1,1,2,2-tetrafluoroethane (CFC 114)	TO-15	76-14-2	0.19	0.5	ug/m3					65	-	122		25
Vinyl Chloride	TO-15	75-01-4	0.17	0.5	ug/m3					65	-	128		25
1,3-Butadiene	TO-15	106-99-0	0.22	0.5	ug/m3					62	-	143		25
Bromomethane	TO-15	74-83-9	0.19	0.5	ug/m3					65	-	130		25
Chloroethane	TO-15	75-00-3	0.17	0.5	ug/m3					69	-	126		25
Ethanol	TO-15	64-17-5	0.80	5	ug/m3					57	-	126		25
Acetonitrile	TO-15	75-05-8	0.18	0.5	ug/m3					51	-	134		25
Acrolein	TO-15	107-02-8	0.170	2	ug/m3					55	-	146		25
Acetone	TO-15	67-64-1	0.77	5	ug/m3					57	-	120		25
Trichlorofluoromethane	TO-15	75-69-4	0.17	0.5	ug/m3					59	-	139		25
2-Propanol (Isopropyl Alcohol)	TO-15	67-63-0	0.42	5	ug/m3					59	-	129		25
Acrylonitrile	TO-15	107-13-1	0.17	0.5	ug/m3					64	-	136		25
1,1-Dichloroethene	TO-15	75-35-4	0.17	0.5	ug/m3					72	-	123		25
Methylene Chloride	TO-15	75-09-2	0.17	0.5	ug/m3					63	-	117		25
3-Chloro-1-propene (Allyl Chloride)	TO-15	107-05-1	0.16	0.5	ug/m3					50	-	141		25
Trichlorotrifluoroethane	TO-15	76-13-1	0.17	0.5	ug/m3					68	-	118		25
Carbon Disulfide	TO-15	75-15-0	0.15	5	ug/m3					55	-	143		25
trans-1,2-Dichloroethene	TO-15	156-60-5	0.19	0.5	ug/m3					69	-	129		25
1,1-Dichloroethane	TO-15	75-34-3	0.16	0.5	ug/m3					66	-	122		25
Methyl tert-Butyl Ether	TO-15	1634-04-4	0.17	0.5	ug/m3					55	-	128		25
Vinyl Acetate	TO-15	108-05-4	0.65	5	ug/m3					66	-	140		25
2-Butanone (MEK)	TO-15	78-93-3	0.21	5	ug/m3					62	-	127		25
cis-1,2-Dichloroethene	TO-15	156-59-2	0.16	0.5	ug/m3					65	-	125		25
Ethyl Acetate	TO-15	141-78-6	0.35	1	ug/m3					64	-	132		25
n-Hexane	TO-15	110-54-3	0.15	0.5	ug/m3					58	-	126		25
Chloroform	TO-15	67-66-3	0.17	0.5	ug/m3					68	-	117		25
Tetrahydrofuran (THF)	TO-15	109-99-9	0.20	0.5	ug/m3					64	-	123		25
1,2-Dichloroethane	TO-15	107-06-2	0.16	0.5	ug/m3					63	-	124		25
1,1,1-Trichloroethane	TO-15	71-55-6	0.17	0.5	ug/m3					68	-	120		25
Benzene	TO-15	71-43-2	0.16	0.5	ug/m3					61	-	110		25
Carbon Tetrachloride	TO-15	56-23-5	0.15	0.5	ug/m3					65	-	137		25
Cyclohexane	TO-15	110-82-7	0.29	1	ug/m3					68	-	122		25
1,2-Dichloropropane	TO-15	78-87-5	0.16	0.5	ug/m3					67	-	122		25
Bromodichloromethane	TO-15	75-27-4	0.15	0.5	ug/m3					71	-	124		25
Trichloroethene	TO-15	79-01-6	0.14	0.5	ug/m3					71	-	121		25
1,4-Dioxane	TO-15	123-91-1	0.16	0.5	ug/m3					67	-	122		25
Methyl Methacrylate	TO-15	80-62-6	0.31	1	ug/m3					76	-	130		25
n-Heptane	TO-15	142-82-5	0.17	0.5	ug/m3					67	-	125		25
cis-1,3-Dichloropropene	TO-15	10061-01-5	0.14	0.5	ug/m3					73	-	131		25
4-Methyl-2-pentanone	TO-15	108-10-1	0.16	0.5	ug/m3					66	-	132		25
trans-1,3-Dichloropropene	TO-15	10061-02-6	0.16	0.5	ug/m3					76	-	135		25
1,1,2-Trichloroethane	TO-15	79-00-5	0.16	0.5	ug/m3					73	-	121		25
Toluene	TO-15	108-88-3	0.17	0.5	ug/m3					67	-	117		25
2-Hexanone	TO-15	591-78-6	0.16	0.5	ug/m3					59	-	128		25
Dibromochloromethane	TO-15	124-48-1	0.16	0.5	ug/m3					73	-	132		25

UMP023013

Table 1e
Analytical Parameters, Methods and Quantitation Limits
ALS Semi Valley, Air
UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD			
						Recovery			%RPD	Recovery			%RPD
1,2-Dibromoethane	TO-15	106-93-4	0.16	0.5	ug/m3					73	-	128	25
n-Butyl Acetate	TO-15	123-86-4	0.16	0.5	ug/m3					61	-	136	25
n-Octane	TO-15	111-65-9	0.18	0.5	ug/m3					67	-	124	25
Tetrachloroethene	TO-15	127-18-4	0.14	0.5	ug/m3					65	-	126	25
Chlorobenzene	TO-15	108-90-7	0.16	0.5	ug/m3					68	-	120	25
Ethylbenzene	TO-15	100-41-4	0.16	0.5	ug/m3					69	-	123	25
m,p-Xylenes	TO-15	179601-23-1	0.30	1	ug/m3					67	-	125	25
Bromoform	TO-15	75-25-2	0.15	0.5	ug/m3					68	-	153	25
Styrene	TO-15	100-42-5	0.15	0.5	ug/m3					68	-	132	25
o-Xylene	TO-15	95-47-6	0.15	0.5	ug/m3					67	-	124	25
n-Nonane	TO-15	111-84-2	0.15	0.5	ug/m3					60	-	130	25
1,1,2,2-Tetrachloroethane	TO-15	79-34-5	0.15	0.5	ug/m3					72	-	128	25
Cumene	TO-15	98-82-8	0.15	0.5	ug/m3					67	-	124	25
alpha-Pinene	TO-15	80-56-8	0.14	0.5	ug/m3					67	-	129	25
n-Propylbenzene	TO-15	103-65-1	0.16	0.5	ug/m3					67	-	125	25
4-Ethyltoluene	TO-15	622-96-8	0.16	0.5	ug/m3					66	-	128	25
1,3,5-Trimethylbenzene	TO-15	108-67-8	0.16	0.5	ug/m3					65	-	125	25
1,2,4-Trimethylbenzene	TO-15	95-63-6	0.15	0.5	ug/m3					62	-	134	25
Benzyl Chloride	TO-15	100-44-7	0.11	0.5	ug/m3					74	-	145	25
1,3-Dichlorobenzene	TO-15	541-73-1	0.15	0.5	ug/m3					63	-	133	25
1,4-Dichlorobenzene	TO-15	106-46-7	0.14	0.5	ug/m3					62	-	129	25
1,2-Dichlorobenzene	TO-15	95-50-1	0.15	0.5	ug/m3					62	-	134	25
d-Limonene	TO-15	5989-27-5	0.14	0.5	ug/m3					66	-	137	25
1,2-Dibromo-3-chloropropane	TO-15	96-12-8	0.099	0.5	ug/m3					71	-	147	25
1,2,4-Trichlorobenzene	TO-15	120-82-1	0.16	0.5	ug/m3					60	-	145	25
Naphthalene	TO-15	91-20-3	0.18	0.5	ug/m3					56	-	158	25
Hexachlorobutadiene	TO-15	87-68-3	0.14	0.5	ug/m3					56	-	139	25

TO-13A

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD			
						Recovery			%RPD	Recovery			%RPD
Naphthalene	TO-13A	91-20-3		1	ug/sample	60	-	120	30	60	-	120	30
Acenaphthylene	TO-13A	208-96-8		0.5	ug/sample	60	-	120	30	60	-	120	30
Acenaphthene	TO-13A	83-32-9		0.5	ug/sample	60	-	120	30	60	-	120	30
Fluorene	TO-13A	86-73-7		0.5	ug/sample	60	-	120	30	60	-	120	30
Phenanthrene	TO-13A	85-01-8		0.5	ug/sample	60	-	120	30	60	-	120	30
Anthracene	TO-13A	120-12-7		0.5	ug/sample	60	-	120	30	60	-	120	30
Fluoranthene	TO-13A	206-44-0		0.5	ug/sample	60	-	120	30	60	-	120	30
Pyrene	TO-13A	129-00-0		0.5	ug/sample	60	-	120	30	60	-	120	30
Benz(a)anthracene	TO-13A	56-55-3		0.5	ug/sample	60	-	120	30	60	-	120	30
Chrysene	TO-13A	218-01-9		0.5	ug/sample	60	-	120	30	60	-	120	30
Benzo(b)fluoranthene	TO-13A	205-99-2		0.5	ug/sample	60	-	120	30	60	-	120	30
Benzo(k)fluoranthene	TO-13A	207-08-9		0.5	ug/sample	60	-	120	30	60	-	120	30
Benzo(a)pyrene	TO-13A	50-32-8		0.5	ug/sample	60	-	120	30	60	-	120	30
Indeno(1,2,3-cd)pyrene	TO-13A	193-39-5		0.5	ug/sample	60	-	120	30	60	-	120	30
Dibenz(a,h)anthracene	TO-13A	53-70-3		0.5	ug/sample	60	-	120	30	60	-	120	30
Benzo(g,h,i)perylene	TO-13A	191-24-2		0.5	ug/sample	60	-	120	30	60	-	120	30

TO-17 Tenax Tube

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD			
						Recovery			%RPD	Recovery			%RPD
Naphthalene	TO-17	91-20-3	0.19	0.50	ng/tube					70	-	130	25
1-Methylnaphthalene	TO-17	90-12-0	0.13	0.50	ng/tube					70	-	130	25
2-Methylnaphthalene	TO-17	91-57-6	0.081	0.50	ng/tube					70	-	130	25
Acenaphthylene	TO-17	208-96-8	0.13	0.50	ng/tube					70	-	130	25
Acenaphthene	TO-17	83-32-9	0.072	0.50	ng/tube					70	-	130	25

UMP023014

Table 1e
Analytical Parameters, Methods and Quantitation Limits
ALS Semi Valley, Air
UMore Park/Former Gopher Ordnance Works Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	EPA Method	CAS#	MDL	RL	Unit	MS / MSD				LCS / LCSD			
						Recovery			%RPD	Recovery			%RPD
Fluorene	TO-17	86-73-7	0.14	0.50	ng/tube					70	-	130	25
Phenanthrene	TO-17	85-01-8	0.34	0.50	ng/tube					70	-	130	25
Anthracene	TO-17	120-12-7	0.38	0.50	ng/tube					70	-	130	25
Fluoranthene	TO-17	206-44-0	0.32	1.0	ng/tube					70	-	130	25
Pyrene	TO-17	129-00-0	0.34	1.0	ng/tube					70	-	130	25

(1) The laboratory reporting limits (RL) and minimum detection limits (MDL) are periodically updated. The RL and MDL may vary based on initial volume and possible matrix interferences.

(2) The laboratory LCS/LCSD, MS/MSD spike recoveries and RPDs are periodically updated. The LCS/LCSD and MS/MSD spike recoveries and RPDs noted in the laboratory report will be used for data validation.

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**PREPARATION OF AQUEOUS SAMPLES FOR TESTING BY ICP (200.7-6010C)**
SOP NO.:	**LABENV-042.10**

Original Information			
Prepared by:	William R. Dahl	Date:	01/30/02
Technical Review:	Brian Leigh	Date:	02/26/02
QA/QC:	Terri Olson	Date:	02/26/02
Authorized by:	Cheryl Sykora	Date:	02/26/02

Revision Information			
Supersedes:	LABENV-042.9	Date:	02/23/15
Revised by:	Ryan Hill		
Signature:	Electronic Signature/Signature Date on File		
Technical Review:	Kelleigh Wasser		
Signature:	Electronic Signatures/Signature Date on File		
QA/QC:	Amy Ostergren		
Signature:	Electronic Signature/Signature Date on File		
Authorized by:	Amy Ostergren		
Signature:	Electronic Signature/Signature Date on File		

SOP TITLE: PREPARATION OF AQUEOUS SAMPLES FOR TESTING BY ICP (200.7-6010C)

1. PURPOSE

1.1 This document defines the preparation of samples prior to analysis by ICP. It applies to all aqueous matrices and to terminology referring to "liquids" and "TCLP extracts/leachates." The SOP is applicable to samples typically prepared by EPA 200.7, EPA 6010C, and EPA 3005A.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance representative to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in this SOP.

2.3 When this method is used for controlled substance sample preparation, personnel performing this method must be authorized per the LEGEND Controlled Substance Program.

2.4 Analysts trained by Legend Technical Services, Inc. (LEGEND) shall perform analyses. Training requirements are outlined in LEGEND's training program for the method. After training and prior to performing this procedure, each new analyst must successfully complete an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is appropriate to aqueous liquids with a density around 1 g/mL. If a sample does not fit this criterion it must be prepared using a solid preparation method and reported on a weight basis.

3.2 Nitric acid preservation of samples for dissolved metals analyses is performed to assist with separating metals from the matrix and avoid redisposition.

3.3 Controlled substance samples are located in the secure controlled substance area. Collection and weighing/aliquoting of the subsample for analysis must be performed in this area. It is recommended that samples are directly weighed/placed into their designated preparation container.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The samples shall be accepted or rejected if/if not packaged to protect the sample's integrity and clearly/not clearly labeled for identification per the 'Sample Receiving, Handling, Log-In, Storage Control & Holding Times' SOP.

5.2 Controlled substance samples shall be accepted/rejected per the requirements of LEGEND's Controlled Substance Program.

5.3 Water samples should be collected in polyethylene or glass containers and preserved with 1:1 nitric acid to a pH < 2. **NOTE**: If the laboratory will be filtering the sample for dissolved metals analyses, do not perform nitric acid preservation.

5.4 The recommended holding time for water samples is six months.

5.5 If a sample is received with pH > 2 then it is preserved in the laboratory and allowed to sit for a minimum of 24 hours at which time the pH is re-tested. If pH < 2, proceed. If not, repeat until pH is < 2, if possible. Highly alkaline samples may need to be digested at reduced volume with excess acid, rather than dilute the original sample in an attempt to lower the pH.

5.6 Document the final pH of all samples on the raw bench sheet. For samples requiring preservation at the laboratory, document the addition of acid and the time/date of addition and the time of re-checking the pH in the pH Adjustment of Samples for Metals Analyses logbook located in the metals lab. Also record the LIMS ID of Acid used and the volume used.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Environmental Express Hotblock Model SC154, or equivalent

6.2 50 mL Screw cap digestion vessels, certified for volume, Environmental Express SC475 or equivalent (Records kept in the QA/QC department)

6.3 Disposable plastic watch glass covers, Environmental Express SC505, or equivalent

6.4 Nitric acid (HNO_3), Trace Metal Grade Fisher A509212

6.5 Hydrochloric Acid (HCl), Trace Metal Grade, Fisher A508212

6.6 Bottle top dispensers for acids

6.7 0.45 µm, Plunge filters, Environmental Express SC0401 or equivalent

6.8 ICP Standard Stock Solutions: Inorganic Ventures #LTS-STOCK-1A, #LTS-STOCK-2A, #LTS-STOCK-3A and #MSHG-100PPM or equivalent.

6.9 Deionized (DI) water (>16.3 MΩ)

7. PROCEDURE

7.1 Prepare a LIMS sample batch with required QC samples (BLK, LCS, LCSD, MS, and MSD). Print out a bench sheet. A batch may contain up to 20 samples for 6010C and up to 20 samples for 200.7. A MS/MSD is required for every 10 samples for 200.7 analysis.

7.2 Indicate which source sample is used, as well as which standards, for spiking and reagent numbers on the bench sheet.

7.3 Batch TCLP, 200.7, and 6010C samples separately, i.e. all TCLP samples in one batch, 200.7 samples in one batch and 6010 samples in another.

7.4 Label digestion vessels with sample IDs, including the Blank, MRL, MS, MSD, LCS, and LCSD samples. Make sure to include the batch number. Labels printed from LIMS may be used.

7.5 Add 0.050 mL of spike solution from each of the first three groups listed below and 0.25 mL from the fourth group to the LCS, LCSD, MS, and MSD (final volume will be 50 mL) for all batch types except TCLP. For TCLP batches, add 0.10 mL of spike solution from each of the first three groups and 0.25 mL from the fourth group listed below.

Spike Group 1

Element	Stock Concentration (ppm)	Final Concentration (ppm)	TCLP Final Concentration (ppm)
Aluminum	2000	2.0	4.0
Barium	400	0.40	0.80
Calcium	4000	4.0	8.0
Cobalt	400	0.40	0.80
Iron	2000	2.0	4.0
Magnesium	4000	4.0	8.0
Manganese	400	0.40	0.80
Potassium	2000	2.0	4.0
Sodium	4000	4.0	8.0
Vanadium	400	0.40	0.80
Zinc	400	0.40	0.80

Spike Group 2

Arsenic	400	0.40	0.80
Beryllium	40	0.040	0.080
Cadmium	400	0.40	0.80
Chromium	400	0.40	0.80
Copper	400	0.40	0.80
Lead	400	0.40	0.80
Nickel	400	0.40	0.80
Selenium	400	0.40	0.80
Silver	40	0.040	0.080
Thallium	400	0.40	0.80

Spike Group 3

Antimony	400	0.40	0.80
Boron	400	0.40	0.80
Molybdenum	400	0.40	0.80
Tin	400	0.40	0.80

Spike Group 4

Mercury	100	0.25	0.50

7.6 Shake all samples prior to transferring into digestion vessels.

7.7 For all batch types except TCLP, add DI water to the 50 mL mark on the digestion vessels for each of the Blank, MRL, LCS, and LCSD. For TCLP batches, add 10 mL of the TCLP leachate blank to each of the Blank, LCS and LCSD and then dilute to the 50 mL mark with DI water.

7.8 For all samples except TCLP, measure out 50 mL of sample directly into its assigned digestion vessel as well for the digestion vessels designated for the MS and MSD. For TCLP samples, measure out 10 mL of the TCLP sample into the designated digestion vessel and dilute to the 50 mL mark with DI water as well as for the digestion vessels designated for the MS and MSD.

7.9 Alternate volumes may be used if sample availability is limited or a dilution is required.

7.10 Record the initial volumes of all samples on the bench sheet.

7.11 Transfer the samples to a sample holding rack.

7.12 For 6010C samples, add 2 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl to all the samples (including Blank, LCS, & LCSD). For 200.7 samples add 1 mL of 1:1 HNO_3 and 0.5 mL 1:1 HCL.

7.13 Ensure that the internal temperature of an equilibrated DI water digestion vessel sample reads between 95 – 97ºC prior to using the hotblock digester. (Note: The temperature of the hotblock itself may need to be adjusted to ensure the temperature requirement is met.) Record the temperature of the sample, along with the thermometer ID, on the bench sheet.

7.14 Transfer samples from the holding racks to the hotblock digester and place plastic watch glasses over each digestion vessel. Record the time the samples are placed into the hotblock digester.

7.15 Let samples heat for 2 to 4 hours, ensuring sufficient volume loss.

7.16 Remove samples from hotblock digester and allow samples to cool to room temperature. Record time removed from the hotblock digester.

7.17 Bring all samples up to 50 mL with DI water using the graduations on the digestion vessels.

7.18 Record the final volumes on the bench sheet.

7.19 Filter only those samples where sediment is present using the plunge filters. If samples must be filtered, you must also filter the QC, including the Blank, LCS, LCSD, MS and MSD. If samples are not being filtered, do not filter the QC samples.

7.20 Cap all samples and invert to mix.

7.21 Place samples sample holding racks.

7.22 Calibration, analysis and calculations are not applicable to this SOP but are addressed in the 'Analysis of Samples by Axial ICP- (200.7-6010C)'SOP.

7.23 Any prepared controlled substance sample remaining after analysis may be refrigerated for up to 30 days.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures. Refer to analytical testing SOP for disposal requirements.

8.1 Highly contaminated samples may be returned to the client for disposal.

8.2 Return all remaining controlled substance sample extracts to the secure controlled substance location when testing is complete. Log them back into the sample inventory as required per the Controlled Substance Program.

9. QA/QC

9.1 Follow the QA/QC protocol outlined in the 'Analysis of Samples by Axial ICP-AES (200.7-6010C)' SOP.

10. REPORTING

Not applicable

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

12. REFERENCES

12.1 Method EPA 200.7, Revision 4.4, May 1994

12.2 Method EPA 3005A, Revision 1, Jul7 1992

12.3 Method EPA 6010C, Revision 3, February 2007

12.4 LEGEND SOP 'Analysis of Samples by Axial ICP-AES (200.7-6010C)'

12.5 LEGEND Controlled Substance Program

Appendix A

Initial Demonstration of Capability (IDC)
Preparation and/or Analysis of Aqueous Samples for ICP (200.7-6010C)

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. If performing the analysis portion of the IDC, prepare a calibration curve in addition to a solvent/method blank and four mid-level laboratory control standard samples in lab-grade water.

3. Analyze the calibration curve, the solvent/method blank, and samples per the SOP.

4. Enter individual data results using LEGEND form 'IDC, 4 Repl, % RPD-Relative % Difference between Min & Max'. The form will calculate the mean recovery in concentration and %, and the % RPD of the replicates.

5. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results, including QC data

6. The results must meet the following criteria:

   - QA/QC: All QA samples must meet requirements of the method
   - Accuracy: EPA 200.7, Within current limits (85 – 115%) for analyte recovery
EPA 6010C, Within current limits (80 – 120%) for analyte recovery
NOTE: 200.7/6010B may be combined if 200.7 limits are used
   - Precision: Within limits (≤ 20%) for each analyte
   - Reagent blank: Must be less than the current reporting limit (RL) for each analyte

7. If the sample or QC data does not meet requirements (i.e. is not acceptable), the IDC must be repeated.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC representative sign and file the training documentation as required per LEGEND's training program.

DOCUMENT REVIEW

DOCUMENT:	LABENV-042.10
REVIEWER:	Kelleigh Wasser
DATE:	03/04/16

SECTION	CHANGE	RATIONALE
LABENV-042.10		
5.6	Added the use of a logbook to samples that require preservation at the laboratory and updated documentation requirements	Record keeping and good practice
7.1	Changed 10 to 20 samples for 200.7 and added MS/MSD requirement	Efficiency/Method Requirement
7.1.1, 7.1.12	Removed	Not current practice
LABENV-042.9		
Cover Page, footers	Revised cover page to new format and for documented change revision/approval in electronic quality management system. Updated footers.	New form includes QA sign-off and Implementation of electronic quality management system.
Cover Page, Title	Added method references.	Missing.
Entire document	Added method 6010C.	Missing from scope.
2.1	Updated supervisor to representative.	Expand scope of personnel.
2.3, 3.3, 5.2, 8.3	Added controlled substance sample requirements.	Expansion of method scope.
2.4	Updated training definition.	Result of formalization of Legend training program.
3.2	Updated reason for nitric acid preservation.	Clarification.
4.3	Removed.	Considered part of standard lab requirements.
5.1	Updated and combined sample acceptance/rejection criteria and sample storage criteria.	Updated SOPs and CRF requirements.
6	Updated reagents and supplies with part numbers and better descriptions. Added missing standard information.	Clarification.
7	Minor revisions to wording throughout section.	Clarification and document flow.
7.1	Added missing 200.7 MRL and turbidity use.	Expansion of method scope.
7.4	Added use of labels printed from LIMS.	Current lab practice.
7.5	Addition of mercury to the spike solutions.	Expansion of method scope.
7.13	Updated the description for hotblock temperature setting.	Clarification.
7.15	Removed requirement for volume loss.	Not necessary for this process.
7.22, 9.1	Added specific method.	Clarification.

DOCUMENT REVIEW

DOCUMENT:	LABENV-042.10
REVIEWER:	Kelleigh Wasser
DATE:	03/04/16

SECTION	CHANGE	RATIONALE
7.23	Added retention, stability statement for prepared controlled substance samples.	Expansion of method scope.
8.2	Added statement about returning highly contaminated samples to clients for disposal.	LEGEND policy.
12	Updated/added EPA method references.	Missing references.
12	Remove references to EPA 6010B.	No longer accredited for 6010B.
Appendix A	Updated content of IDC to match training requirements and new/upcoming requirements of the AIHA policies.	Instituted LEGEND training program. AIHA program update.
LABENV-042.8		
Entire document	Removed reference to AA.	No longer used
6.2	Updated digestion vessel to certified.	Currently used
7	Slight rewording, reorganization for document clarity and flow.	Clarification
7.4, 7.19	Added MS and MSD.	Current practice
7.5	Spike preparation moved up/earlier in document to match actual prep.	Current practice
7.5	Added column to table for TCLP spike concentrations.	Clarification
7.13	Added to record internal tube temperature of a blank sample.	Audit response
7.15	Defined sufficient volume.	Clarification
7.16	Added to record time removed from digester.	Record completeness
12.2	Added reference to 6010C.	New revision available
LABENV-042.7		
7.3	TCLP, 200.7 and 6010 batched separately	Current practice
7.14	Added amount of acid to add specific for 200.7	MDH Audit

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**ANALYSIS OF SAMPLES BY AXIAL ICP-AES (200.7, 6010C)**
SOP NO.:	**LABENV-039.11**

Original Information			
Prepared by:	William Dahl	Date:	05/24/00
Technical Review:	Name	Date:	05/24/00
QA/QC:	NA	Date:	NA
Authorized by:	Cheryl Sykora	Date:	09/28/00

Revision Information			
Supersedes:	**LABENV-039.10**	Date:	02/23/15
Revised by:	Ryan Hill		
Signature:	Electronic Signature/Signature Date on File		
Technical Review:	Kelly French		
Signature:	Electronic Signatures/Signature Date on File		
QA/QC:	Sarah Smestad		
Signature:	Electronic Signature/Signature Date on File		
Authorized by:	Cheryl Sykora		
Signature:	Electronic Signature/Signature Date on File		

SOP TITLE: ANALYSIS OF SAMPLES BY AXIAL ICP-AES (200.7, 6010C)

1. PURPOSE

1.1 This document defines the analysis procedure for various metals by axially viewed inductively coupled plasma atomic emission spectroscopy (ICP-AES). The SOP is applicable to samples typically analyzed by EPA 200.7, EPA 6010C, and a modified NIOSH 7303.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance representative to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in this standard operating procedure.

2.3 When this method is used for controlled substance sample preparation, personnel performing this method must be authorized per the LEGEND Controlled Substance Program.

2.4 Analysts trained by Legend Technical Services, Inc. (LEGEND) shall perform analyses. Training requirements are outlined in LEGEND's training program for the method. After training and prior to performing this procedure, each new analyst must successfully complete an Initial Demonstration of Capability (IDC) prior to analyzing samples. The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is liquid matrices and is applicable to digestates of various matrices.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The samples shall be accepted or rejected if/if not packaged to protect the sample's integrity and clearly/not clearly labeled for identification per the 'Sample Receiving, Handling, Log-In, Storage Control & Holding Times' SOP.

5.2 Controlled substance samples shall be accepted/rejected per the requirements of LEGEND's Controlled Substance Program.

5.3 Water samples should be collected in polyethylene or glass containers and preserved with 1:1 nitric acid to a pH < 2. These samples may be stored ambient. Samples to be filtered in the laboratory should be collected in an unpreserved bottle. Unpreserved samples should be cooled and stored at ≤ 6^{o} C, but not freezing, until filtered in the laboratory.

5.4 If a water sample is received with pH > 2, it is preserved in the laboratory and allowed to sit for a minimum of 24 hours at which time the pH is re-tested. If pH < 2, proceed with testing. If not, repeat preservation until pH is < 2 if possible. Highly alkaline samples may need to be digested at reduced volume with excess acid, rather than dilute the original sample in an attempt to lower the pH.

5.5 Document pH and/or lab filtration on a raw data batch/bench sheet. Document the addition of acid and the time acid was added in the sample comments and work order comments in the work order section of the LIMS.

5.6 The recommended holding time for water samples is six months.

5.7 Solid environmental samples should be collected in polyethylene or glass containers and stored at ≤ 6°C, but not freezing, then moved to ambient conditions when testing is complete and results reviewed.

5.8 The recommended holding time for solid samples is six months.

5.9 Medical device leachates are analyzed as received, unless otherwise noted by the project.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Varian Model VISTA AX™ ICP-AES Instrument, or equivalent

6.2 Varian Model SPS-5™ Autosampler, or equivalent

6.3 Assorted laboratory glassware

6.4 15 mL disposable centrifuge tubes (used for dilutions)

6.5 Nitric acid (HNO_3), Trace Metal Grade, Fisher A509212

6.6 Hydrochloric acid (HCl), Trace Metal Grade, Fisher A508212

6.7 De-ionized (DI) water (>16.3 MΩ)

6.8 Single element stock standards: Inorganic Ventures, or equivalent

6.9 Calibration Stock Standards: Inorganic Ventures #LTS-STOCK-1A, #LTS-STOCK-2A, and #LTS-STOCK-3A, #MSHG-100PPM or equivalent

6.10 Second Source Stock Standards: second source supplier or lot number different than that of calibration stock standards: High Purity #SM-3705-001 (Solutions A and B), Inorganic Ventures #CGBA1, #CGBI1-1, #CGSE(4)1, #MSHG-100PPM, #CGSR1-1 or equivalent

6.11 Interference standards: Inorganic Ventures #CGFE10, #CGAL10, #CGCA10, and #CGMG,10 or equivalent Low-level check standards: Environmental Express HP3201-250 Solutions A and B, Inorganic Ventures #MSHG-100PPM, #CGSINA1-1 or equivalent

6.12 Liquid Argon Dewars

6.13 10,000 ppm lead standard, Inorganic Ventures CGPB10-1 or equivalent

6.14 100 ppm mercury standard, Inorganic Ventures MSHG-100PPM or equivalent

7. PROCEDURE

7.1 Preparation of Samples

7.1.1 Aqueous samples are prepared for analysis using the SOP ‘Preparation of Aqueous Samples for Testing by ICP (200.7-6010C)’ and ‘Static and Kinetic Testing of Mining Waste Rock and Ore (humidity cells)’.

7.1.2 Soil/solid samples are prepared for analysis using the SOP ‘Preparation of Solid Samples for Testing by ICP (6010C)’.

7.1.3 For AIHA samples (air, paint, wipes, and soil samples for ELPAT) refer to SOP 'Preparation of Paint, Air, and Dust Wipe Samples for Metals Analysis (EPA 6010C, NIOSH 7303)'.

7.1.4 Medical device leachates samples do not require digestion. Due to the matrix effect on the internal standards, saline and phosphate buffered leachates are diluted by a factor of two and analyzed. DI leachates can be analyzed with no dilution. Other matrices may be analyzed as well. Appropriate dilutions will be made to ensure internal standards fall within acceptable ranges. Due to limited sample typically received, only an MS is analyzed instead of both an MS and MSD.

7.2 Calibration

7.2.1 Refer to Equipment SOP 'Inductively Coupled Plasma – Atomic Emission Spectrometer' SOP for instrument set-up.

7.2.2 Calibration for all metals, except lead, consists of one calibration standard prepared in 4% HNO_3 and 5% HCl from the Calibration Stock Standards and a calibration blank. Typical calibration concentrations are:

Element	Level (ppm)	Element	Level (ppm)	Element	Level (ppm)
Ag	0.40	Cr	4.0	Hg	2.0
Al	20	Cu	4.0	S	8.0
As	4.0	Fe	20	Sb	4.0
B	4.0	K	20	Se	4.0
Ba	4.0	Li	4.0	SiO2	8.0
Be	0.40	Mg	40	Sn	4.0
Ca	40	Mn	4.0	Ti	4.0
Cd	4.0	Na	40	Tl	4.0
Sr	4.0	Ni	4.0	V	4.0
Co	4.0	P	4.0	Zn	4.0

7.2.3 For lead, calibration is performed using five concentration levels prepared at 0.02ppm, 0.2ppm, 2.0ppm, 20.0ppm and 200.0ppm.

7.2.4 Alternative calibration levels may be used to meet project specific requirements

7.2.5 Analyze calibration standards with conditions outlined in the equipment 'Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES)' SOP, using the standards and frequencies outlined in the analysis section below in this SOP.

7.2.6 Evaluate the calibration:

7.2.6.1 Linearity: For all metals except lead, calibration is a single point linear calibration through zero. There is no linearity requirement for those metals. For lead, construct a calibration curve using Concentration vs. Response with a first order or linear fit. The correlation coefficient (r^2) must be 0.998 or greater.

7.2.6.1.1 If the correlation coefficient (r2) is ≥ 0.998, the calibration is considered acceptable and may be used for quantitation of that compound.

7.2.6.1.2 If the correlation coefficient (r^2) is less than 0.998, corrective action should be taken.

7.2.6.2 Calibration accuracy: For EPA 6010C, the ICV / CCV recoveries must be 90 – 110%. For EPA 200.7, the QCS / first IPC (CCV) recoveries must be 95 – 105% and the subsequent IPC / CCV recoveries must be 90 – 110%. If not met corrective action must be taken.

7.2.6.3 Reporting limit verification: Analyze a reporting limit verification standard (RLV) with each calibration curve by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit. **NOTE**: An RLV check must also be performed at the end of a sequence for EPA 6020A analyses. The acceptance criteria must meet the following or corrective action must be taken:

7.2.6.3.1 EPA 200.7: 60 – 140%

7.2.6.3.2 EPA 6010C: 70 – 130%

7.2.6.3.3 AIHA (ELLAP): 80 – 120% (also called the CRDL)

7.2.6.4 Corrective action may include reanalyzing the standard, preparing and analyzing a new standard, preparing a new calibration curve, and/or using new reagents, performing maintenance, recalibrating, and/or raising the reporting limit.

7.2.6.5 Calibration curve calculations are found in the 'Quality Assurance Parameters' guidance document.

7.2.7 Metals other than those listed specifically in this SOP may be analyzed by this method as long as all quality requirements are met.

7.3 Analysis

7.3.1 During analysis, samples with results greater than 90% of the LDR are diluted and re-analyzed.

7.3.2 Method EPA 6010C and AIHA (ELLAP) Standards and QC Requirements

7.3.2.1 Initial Calibration Verification (ICV): This is a multi-element standard (including lead) prepared in 4% HNO_3 and 5% HCl from Second Source Stock Metals Standard(s). Analyze immediately after the calibration. Recovery must be within $\pm$ 10% of the true value. If this fails, any data generated is invalid. Corrective action should be taken and the analysis repeated.

7.3.2.2 Continuing Calibration Verification (CCV): This is a multi-element, mid-level standard (including lead) prepared in 4% HNO_3 and 5% HCl from Calibration Stock Metals Standard(s). Analyze following the calibration, after every 10 samples, and at the end of the run. Recovery for elements of interest must within $\pm$ 10% of the true value. If not, samples bracketed by an out-of-spec CCV must be re-analyzed. **NOTE**: If reanalysis is not possible and sample results must be reported with an out-of-spec CCV due to limited sample volume or other unusual circumstance, the data will be qualified and a method non-conformance (MNC/NCP) initiated.

7.3.2.3 Initial and Continuing Calibration Blanks (ICB, CCB): This is a 4% HNO_3/5% HCl solution in DI water. Analyze following the calibration/at the beginning of the run, after every 10 samples, and at the end of the run. The absolute value of the result for elements of interest must be less than the reporting limit. If not, samples bracketed by an out-of-spec CCB must be re-analyzed. No data for failing elements can be reported. **NOTE**: If sample results must to be reported with an out-of-spec CCB due to limited sample volume or other unusual circumstance the data will be qualified and a method non-conformance (MNC/NCP) initiated.

7.3.2.4 Interference Check Standard A (ICSA or IFA in LIMS): This is a multi-element standard consisting of Fe at 300 mg/L, and Al, Ca, and Mg at 200 mg/L prepared in 4% HNO_3 and 5% HCl prepared from single element stock standards, used to verify the inter-element corrections (IEC) of the analytical method. Analyze after the calibration/at the beginning of each run, before samples, and at the end of the run. The absolute value of the analytes of interest (other than Fe, Al, Ca and Mg) must be less than the value of the reporting limit. If not, evaluate the single element IEC/SIC solutions for any corrections required.

7.3.2.5 Spectral Interference Check (SIC) Solutions: These are single element standards (Fe at 300 mg/L, Al at 200 mg/L, Ca at 200 mg/L, and Mg at 200 mg/L) prepared in 4% HNO_3 and 5% HCl from single element stock standards. The SIC Solutions are used to verify and/or correct other elements for interferences from Fe, Al, Ca and Mg. They are analyzed with every run since Fe, Al, Ca and Mg are the most prevalent sources of interference for environmental samples. If interference is present for an element of interest, re-calculate the IEC factors based on the response from these standards, update the ICP method, and re-process the run to apply these corrections. Refer to Equipment SOP 'Inductively Coupled Plasma – Atomic Emission Spectrometer' for instruction on recalculating and updating IEC factors.

7.3.2.6 Annual Spectral Interference Check (SIC): Annually, single element standards are analyzed to validate IEC factors for all elements (not just Fe, Al, Ca, Mg – these elements are known to have potential spectral overlaps with analytes of interest). The following elements are analyzed: Fe, Al, Ba, Be, Cd, Ce, Co, Cr, Cu, Mn, Ni, Si, Sn, Ti, Tl, V, Zn, Pb, and Mo. If interference is detected, re-calculate the IEC factors and update the ICP method.

7.3.2.7 Annual Linear Dynamic Range (LDR) Determination: Annually (or when a new instrument method is developed), determine the linear dynamic range. Standards are measured at successively increasing concentrations with the acceptance criteria that the standards recover within $\pm$ 10% of the true value.

7.3.3 Method EPA 200.7

7.3.3.1 Quality Control Sample (QCS): This is identical to the ICV standard above in 7.3.2.1. Analyze following the calibration. Recovery must be within $\pm$ 5% of the true value. If this fails for any of the elements of interest, the run must be stopped and the instrument re-calibrated.

7.3.3.2 Instrument Performance Check (IPC/CCV): This is a multi-element (including lead), mid-level standard prepared in 4% HNO_3 and 5% HCl f from Calibration Stock Standard(s). Analyze following the calibration/at the beginning of each run, after every 10 samples, and at the end of the run.

7.3.3.2.1 Recovery of the first/initial IPC (CCV) must be within ± 5% of the true value. If this fails for any of the elements of interest, the run must be stopped and the instrument recalibrated.

7.3.3.2.2 Recovery for continuing IPC (CCV) determinations must be within ± 10% of true value. If this fails for any of the elements of interest, samples bracketed by an out-of spec IPC (CCV) must be re-analyzed following recalibration of the instrument.

7.3.3.3 Calibration Blank Check: This is identical to the CCB/ICB above in 7.3.2.3, prepared and evaluated in the same manner.

7.3.3.4 Interference Check Standard A (IFA): This is identical to the IFA above in 7.3.2.4, prepared and evaluated in the same manner; however, it is also analyzed and evaluated at the end of the run.

7.3.3.5 Spectral Interference Check (SIC) Solutions: This is identical to the SICs above in 7.3.2.5, prepared and evaluated in the same manner.

7.3.3.6 Annual Spectral Interference Check (SIC): This is identical to the annual SIC check above in 7.3.2.6, prepared and evaluated in the same manner.

7.3.3.7 Annual Linear Dynamic Range (LDR) Determination: Preparation and evaluation of the range is identical to 7.3.2.7 above..

7.3.4 For AIHA related work, samples with an in-solution concentration greater than the highest calibration level (for the analyte(s) of interest) must be diluted such that the resulting concentration falls below the highest concentration calibration standard. If not, data must be flagged and qualified.

7.3.5 Export ICP data for the run into the corresponding LIMS sequence.

7.4 Calculations

7.4.1 Calculate the concentration of the analyte in the sample using the following equations:

$$Water\ Concentration\ (mg/L) = \frac{(C_{in})(FV)(D)}{V}$$

$$Soil\ Concentration\ (mg/kg) = \frac{(C_{in})(FV)(D)}{M}$$

C_{in} = in-solution concentration, mg/L
FV = final volume, mL
D = dilution factor
V = volume of sample, mL
M = mass of sample, g

7.5 Data review and Disposition

7.5.1 Evaluate, review and report data as outlined in the 'Documentation Procedures' SOP.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

8.3 Return all remaining controlled substance sample extracts to the secure controlled substance location when testing is complete. Log them back into the sample inventory as required per the Controlled Substance Program.

8.4 Retain samples 30 days after the data is reported. Retain digestates for two months after the preparation date.

8.5 Dispose of samples and digestates in the acid neutralizing laboratory sinks.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven digested replicate spikes per reference method or regulatory program requirements, when there is a change in the test method that may affect how the test is performed, when there is a major change in instrumentation, or at minimum annually.

9.1.2 Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client/program requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Examples of typical MDLs and RLs values can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The absolute value of the method blank must be less than the reporting limit or the sample batch is re-digested. If it is not possible to re-digest, the data will be flagged where appropriate. If data is subsequently flagged, complete a Method Non-Conformance (MNC/NCP) report. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits for laboratory control samples (LCS/LCSD, BS/BSD) and matrix spikes (MS/MSD) are evaluated.

9.3.1 Accuracy control limits for LCS (BS) and MS are set, per the regulatory method requirements, at the following:

LCS:	EPA 200.7	=	75 – 125%
	EPA 6010C	=	80 – 120%
	NIOSH 7303(M)	=	80 – 120%
MS:	EPA 200.7	=	75 – 125%
	EPA 6010C	=	80 – 120%

9.3.2 Precision control limits for LCS/LCSD (BS/BSD) and MS/MSD are set at ≤ 20% RPD.

9.3.3 QC calculations are found in the 'Quality Assurance Parameters' Guidance Document.

9.3.4 LCS (BS) and MS

9.3.4.1 If the LCS data are outside the limits, the sample batch is re-digested. If the batch cannot be re-digested, the data is flagged and a Method Non-Conformance (MNC/NCP) report is filled out.

9.3.4.2 If the LCS has been reviewed and found acceptable and the MS data are outside the limits, the data for that specific MS/ and source sample is flagged and reported as required.

10. REPORTING

10.1 Solid sample results are reported in mg/kg on a dry weight basis unless specified otherwise by the client.

10.2 Bulk sample results are reported in mg/kg on an as received basis unless specified otherwise by the client.

10.3 Water sample results are reported in mg/L unless specified otherwise by the client.

10.4 The reported result is rounded to two significant figures.

10.5 A LIMS generated printout of the results is placed in the project file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A - Initial Demonstration of Capability

11.2 Appendix B - Example of Typical Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 Method EPA 200.7, Revision 4.4, May 1994

12.2 Method EPA 6010C, Revision 3, February 2007

12.3 Method NIOSH 7303, Issue 1, March 2003

12.4 LEGEND SOP 'Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES)'

12.5 LEGEND SOP 'Preparation of Aqueous Samples for Testing by ICP (200.7-6010C)

12.6 LEGEND SOP 'Preparation of Solid Samples for Testing by ICP (6010C)'

12.7 LEGEND Controlled Substance Program

Appendix A

Initial Demonstration of Capability (IDC)
Preparation and/or Analysis of Aqueous Samples for ICP (200.7-6010C)
Preparation and/or Analysis of Solid Samples for ICP (6010C)

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. If performing the analysis portion of the IDC, prepare a calibration curve in addition to a solvent/method blank and four mid-level laboratory control standard samples in lab-grade water.

3. Analyze the calibration curve, the solvent/method blank, and samples per the SOP.

4. Enter individual data results using LEGEND form 'IDC, 4 Repl, % RPD-Relative % Difference between Min & Max'. The form will calculate the mean recovery in concentration and %, and the % RPD of the replicates.

5. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results, including QC data

6. The results must meet the following criteria:

   - QA/QC: All QA samples must meet requirements of the method
   - Accuracy: EPA 200.7, Within current limits (85 – 115%) for analyte recovery
EPA 6010C, Within current limits (80 – 120%) for analyte recovery
NOTE: 200.7/6010B may be combined if 200.7 limits are used
   - Precision: Within limits (≤ 20%) for each analyte
   - Reagent blank: Must be less than the current reporting limit (RL) for each analyte

7. If the sample or QC data does not meet requirements (i.e. is not acceptable), the IDC must be repeated.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC representative sign and file the training documentation as required per LEGEND's training program.

Appendix B

Example of Typical
Method Detection Limits and Reporting Limits
Axial ICP-AES (200.7-6010C)

Element	Water MDL (mg/L)	Water RL (mg/L)	Soil MDL (mg/kg)	Soil RL (mg/kg)
Aluminum	0.0018	0.020	0.090	1.0
Antimony	0.00078	0.010	0.039	0.50
Arsenic	0.0011	0.010	0.055	0.50
Barium	0.0012	0.020	0.060	1.0
Beryllium	0.000028	0.0050	0.0014	0.25
Boron	0.0083	0.10	0.42	5.0
Cadmium	0.00013	0.0010	0.033	0.25
Calcium	0.030	1.0	1.5	50
Chromium	0.00024	0.010	0.012	0.50
Cobalt	0.00033	0.0050	0.017	0.25
Copper	0.0011	0.020	0.055	1.0
Iron	0.0038	0.050	0.19	2.5
Lead	0.00042	0.0030	0.14	1.0
Magnesium	0.0021	1.0	0.11	50
Manganese	0.00078	0.020	0.011	1.0
Molybdenum	0.00070	0.050	0.035	2.5
Nickel	0.00036	0.0050	0.018	0.25
Phosphorous	0.0035	0.050	0.18	2.5
Potassium	0.0016	1.0	0.080	50
Selenium	0.0019	0.020	0.095	1.0
Silver	0.00031	0.0050	0.016	0.25
Sodium	0.0053	1.0	0.27	50
Thallium	0.00057	0.040	0.029	2.0
Tin	0.00059	0.020	0.030	1.0
Titanium	0.0015	0.020	0.075	1.0
Vanadium	0.00016	0.0050	0.0080	0.25
Zinc	0.0036	0.020	0.18	1.0
Mercury	0.0025	0.02	NA	NA

DOCUMENT REVIEW

DOCUMENT:	LABENV-039.11
REVIEWER:	Ryan Hill
DATE:	09/04/15

SECTION	CHANGE	RATIONALE
LABENV-039.11		
Appendix B	Removed non-digestates from table.	Digestion of samples prior to analysis is a method requirement for all elements.
Cover Page	Updated information.	To include Implementation of signatures in electronic quality management system.
LABENV-039.10		
Cover Page, Title	Updated title to include method references.	Missing information.
Entire document	Remove references to EPA 6010B.	No longer accredited for 6010B.
2.3, 5.2, 8.3	Added controlled substance sample requirements.	Expansion of method scope.
2.4	Updated training definition.	Result of formalization of Legend training program.
3.1	Added other liquid matrices.	Expanded scope of SOP.
5.1	Updated and combined sample acceptance/rejection criteria and sample storage criteria.	Updated SOPs and CRF requirements.
6	Updated reagents and supplies with part numbers and better descriptions, including changes to custom standards. Updated with new standards.	Update.
7	Minor revisions to wording throughout section.	Clarification, document flow and harmonization with other ICP methods.
7.2.5	Addition of Equipment SOP reference for analysis set up and conditions.	Missing from SOP.
7.2.6	Update, rearrange calibration standards and evaluation information.	Clarification, document flow and harmonization with other LEGEND methods.
7.3	Removal of mercury limitation for the analysis of only undigested liquid samples.	Expansion of method scope.
7.3.1	Minor updates to standards and QC requirements.	Clarification.
7.5	Removed data review and disposition specifics from SOP.	Method contained within LEGEND's data handling SOP.
9.3, Appendix A	Addition of BS/BSD terminology which is LCS/LCSD for the ICP methods.	Update terminology.
9.3	Update terminology and format for control limits.	Clarification.
9.3.3	Updated QA Manual to QA Parameters document.	Results of QAM update. Missed last update.
12	Updated/added EPA method and other references.	Missing references.

DOCUMENT REVIEW

DOCUMENT:	LABENV-039.11
REVIEWER:	Ryan Hill
DATE:	09/04/15

SECTION	CHANGE	RATIONALE
Appendix A	IDC information revised for requirements.	Result of formalization of Legend training program.
LABENV-039.9		
Cover Page	Revised page to new format or deleted because documented change revision and approval in QT9	New form includes QA sign-off or Implemented in QT9
2.1	Updated supervisor to representative.	Expand scope of personnel.
2.3	Updated training definition.	Result of formalization of Legend training program.
4.3	Removed.	Considered part of standard practices.
5.9, 7.1.4	Added medical device leachates to method.	Expanded scope of SOP.
7.1.1	Added reference to the Mining sample preparation SOP.	Expanded scope of SOP.
7.2.4	Changed QAM to Quality Assurance Parameters Guidance documentation.	Information moved to new reference document.
7.2.6	Added applicability of this method to metals other than those listed in the standard.	Expanded scope of SOP.
7.3.2	Moved location of instruction to dilute samples greater than 90% of the LDR rather than 90% of the highest standard from 7.3.3.7 and 7.3.4.7.	Document flow.
9.2.1, 9.3.4	Expanded definition of nonconformance to include NCP along with MNC.	Expanded documentation system at LEGEND.
Appendix A	Updated content of IDC to match training requirements.	Instituted LEGEND training program.
LABENV-039.8		
Cover Page	Updated to new revision format.	New format to include QA review.
1.1	Added 6010C in addition to 6010B.	Audit response.
5.3	Updated water sample preservation to include storage temperatures.	Audit response.
5.7	Updated 4 ± 2ºC to ≤ 6.0ºC, but not freezing and revised storage directions.	CFR updated definition. Clarification.
6.4, 6.8., 6.10, 6.11	Reworded for clarity the standard information.	Clarification.
6.12	Removed statement about LIMS and traceability	Standard SOP defines this practice.
6.14, 6.15, 6.16	Added 10,000ppm lead standard and 100 and 1000 mercury standards	Standards used by lab
7.1.1-7.1.3	Removed method numbers/added method titles.	Requirement of SOP preparation, to avoid revisions due to potential method number changes.
7.2.2	Added single point concentration for mercury.	Audit response, metals tested by this method but not yet included.

DOCUMENT REVIEW

DOCUMENT:	LABENV-039.11
REVIEWER:	Ryan Hill
DATE:	09/04/15

SECTION	CHANGE	RATIONALE
7.2.3	Added multipoint lead calibration.	Audit response, metals tested by this method but not yet included.
7.2.4	Added project specific calibration levels.	Allows for calibrations outside scope of standard method
7.3.1	Deleted extra standards and QC statement and incorporated into titles of 7.3.2 and 7.3.3	Simplification
7.3.1	Clarified use of method for mercury testing of undigested liquid samples only.	EPA does not recognize mercury testing of digested samples as valid.
7.3.2-7.3.3	Reworded, reorganized definitions of QC samples used for analyses.	Clarification and simplification.
7.3.2,5, 7.3.3.5	Added Mg, and Ca to SIC check.	Current practice.
7.3.4	Deleted IEC statement since it is repetitive from information in previous QC sections.	Simplification.
7.4.2 – 7.4.10	Incorporated into a data review and disposition section as 7.5.	Clarification.
7.5.	Added bench sheets and changed network to company servers.	Clarification.
8.2	Updated sample retention statement.	Clarification to current practice.
9.1.1, 11.2, Appendix B	MDL/RL terminology updated to “Example” MDL/RL.	Current limits being moved to individual files to facilitate availability for use.
9.2	Reorganized wording.	Correct statement.
9.3.4	Move LCS/MS descriptions into this bullet from below it and update wording.	Clarification.
Appendix B	Added mercury to list of metals.	Expanded scope of SOP.
LABENV-039.7		
NA	Updated associated form LABENV-039.7	Annual update
5.7	Specified “environmental” solid samples	Audit response
6.12	Specified stock reagent for RLV check	Audit response
7.2.4	Added 6010C, limits for 6021C RLV and specified when RLV is analyzed.	Expand scope of method
7.3	Added 6010C	Expand scope of method
7.3.2.4	Removed “When possible”	Audit response
7.3.3.4	Removed “When possible”	Audit response

DOCUMENT REVIEW

DOCUMENT:	LABENV-039.11
REVIEWER:	Ryan Hill
DATE:	09/04/15

SECTION	CHANGE	RATIONALE
10.1 – 10.3	Added unless specified otherwise by the client	Client may request other units
12.2	Added 6010C	Expand scope of method

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR USING ACTIVE OR PASSIVE SAMPLING ONTO SORBENT TUBES
REFERENCED METHOD:	EPA TO-17
SOP ID:	VOA-TO17
REV. NUMBER:	09.0
EFFECTIVE DATE:	01/30/2016

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STANDARD OPERATING PROCEDURE

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR USING ACTIVE OR PASSIVE SAMPLING ONTO SORBENT TUBES

EPA TO-17

SOP ID:	VOA-TO17	Rev. Number:	09.0	Effective Date:	01/30/2016

Approved By: _____ Date: 1/25/16
Technical Manager (Volatile GC/MS) - Chris Parnell

Approved By: _____ Date: 1/26/16
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Approved By: _____ Date: 01/26/16
Laboratory Director - Kelly Horiuchi

Archival Date: _____	Doc Control ID#: Non-Controlled	Editor: _____

TABLE OF CONTENTS

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR USING ACTIVE OR PASSIVE SAMPLING ONTO SORBENT TUBES

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in EPA Compendium Method TO-17 and describes a sorbent tube/thermal desorption/gas chromatography-based monitoring method for volatile organic compounds (VOCs) in ambient air at low parts per billion (ppbv) to high parts per million (ppmv) concentration levels. This method is used to quantify a wide range of volatile organic compounds (VOCs) over a wide volatility range.

1.2 The target compound list is the same as listed in Compendium Method TO-15. However, only a portion of these compounds has been monitored by the use of solid adsorbents. Therefore, this method provides performance criteria to demonstrate acceptable performance of the method for monitoring a given compound or set of compounds. Table 2 lists compounds that can be determined by this procedure along with their method reporting limits (MRLs). The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated and documented. Additional compounds may be analyzed according to this procedure as described in the referenced methods as long as the requirements of this document are adhered to; however, if a compound is not listed in the method, it must be reported as a modification.

2) Summary of Procedure

2.1 The monitoring procedure involves pulling a volume of air through a sorbent packing to collect VOCs followed by a thermal desorption GC/MS analytical procedure. The selection of a sorbent or sorbent mix depends on the target compound list, data quality objectives and sampling environment.

2.2 The analytical portion of the method is the same as Compendium Method TO-15 and involves using a high-resolution gas chromatograph (GC) coupled to a mass spectrometer (MS). The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by selective ion monitoring (SIM mode) which consists of monitoring a small number of ions from a specified compound list.

2.3 The sorbent tube is purged with dry, inert gas before analysis to remove any water vapor and air. The sorbent tube is then thermally desorbed (in reverse direction of sampling) onto a low-volume secondary trap where the analytes are refocused. The secondary trap is then rapidly thermally desorbed and the analytes directly transferred onto the head of the GC capillary column. The target analytes are separated by the column using a ramped oven temperature program and detected and measured by mass spectrometry.

2.4 Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with standards and stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response of the primary fragment for known amounts of the compound to establish the concentration that exists in the sample.

3) Definitions

3.1 Sorbent Tube (also referred to as "tube" and "sample tube") Stainless steel, glass lined (or fused silica lined) stainless steel tube, typically ¼ inch O.D. and 3.5 inches long (Perkin Elmer type), with the central portion packed with greater than 200mg of solid adsorbent material, depending on density and packing bed length. The tube is used to concentrate VOCs from air.

3.2 Thermal Desorption The use of heat and flow of inert (carrier) gas to extract volatiles from a solid or liquid matrix directly into the carrier gas and transfer them to downstream system elements such as the analytical column.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 MS-SIM Mass spectrometric mode of operation in which the GC is coupled to an MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

3.5 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.6 Neat Stock Standard A purchased, single component assayed reference material having a stated purity used to prepare working calibration standards.

3.7 Stock Standards Solution A concentrated solution of one or more target analytes at a known concentration purchased from a reputable, approved commercial vendor. Stock standard solutions are used to prepare working calibration standards.

3.8 Intermediate Calibration Standard A solution of one or more target analytes at a known concentration prepared either from one or more neat stock standards or from one or more stock standards solutions.

3.9 Working Calibration Standard A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions.

3.10 Calibration or Standard Curve A calibration or standard curve is a graph that plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.11 Initial Calibration Verification (ICV) Standard A solution prepared in the laboratory containing known concentration(s) of analytes of interest. The solution is prepared from neat stock standards and/or stock standards solutions which are from a different source than the standards used to prepare the working calibration standards.

3.12 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.13 Field Sample A sample collected and delivered to the laboratory for analysis.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop "ticks" have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.15 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Duplicate (LCSD).

3.16 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.17 May This action, activity, or procedural step is neither required nor prohibited.

3.18 Must This action, activity, or procedural step is required.

3.19 Shall This action, activity, or procedural step is required.

3.20 Should This action, activity, or procedural step is suggested, but not required.

3.21 SOP Standard Operating Procedure

3.22 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, sample volume or exposure time, and the service request number (unique number for each submitted job) and serves as an inter-laboratory "custody" form which accompanies all samples throughout the laboratory.

3.23 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or in front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8°C.

3.24 Method Detection Limit (MDL) The MDL is the minimum concentration of a substance or analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

3.25 Limit of Detection (LOD) The Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific. For non-DoD applications LOD = MDL_R. For DoD the LOD is the spike concentration that can be reported with a specific degree of confidence.

3.26 Limit of Quantitation (LOQ) The minimum level, concentration, or quantity of a target analyte that can be reported with a specific degree of confidence.

3.27 Method Reporting Limit (MRL) The minimum level, concentration, or quantity of a target analyte that can be reported with a specific degree of confidence and within the calibration range or equivalent to the low calibration point. The MRL is equivalent to the LOQ.

4) Health and Safety Warnings

4.1 Each compound, mixture of compounds, standards, and surrogates, as well as samples, should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest level possible through the use of gloves (to minimize absorption through the skin) and hoods (to minimize inhalation). Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, SDS location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

4.2 Safety Data Sheets (SDS) The analyst should consult SDS for compounds being handled in the course of this procedure, and be familiar with proper safety precautions to be followed when handling hazardous chemicals. Care should be taken when handling standard material in a neat or highly concentrated form.

4.3 Liquid Nitrogen Liquid nitrogen can cause serious tissue damage (frostbite) with only a few seconds of contact. Valves should be opened slowly so leaky fittings can be identified. Neoprene or leather gloves should be worn when turning valves and handling tubing and fittings that have been in contact with the cryogen.

4.4 Protective Clothing Personal protective clothing (safety glasses, gloves and lab coat) are required when preparing standards and handling standard material in neat form.

4.5 Pressurized Gases The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp when not in use. The regulator should never remain on small "D" size cylinders following use. Sources of flammable gases (i.e. pressurized hydrogen) should be clearly labeled.

4.6 Syringes The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

4.7 Pollution Prevention and Waste Management Samples from tubes are consumed by the analytical procedure, and then the tubes are cleaned and reused. However, any waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.*

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include:

(a) the date of maintenance
(b) who did the maintenance
(c) description of the maintenance
(d) proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, or ordering a part. In addition, a notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

5.2 Automated Thermal Desorber Routine maintenance includes periodic changing of the PTFE-fiber filters and viton o-rings. Filters should be replaced whenever the o-rings are

replaced (fixed-seal and mobile-seal units) and when the cold trap is replaced or removed for maintenance. Under normal usage, this should be a minimum of once every two months. Refer to the TurboMatrix ATD Instrument Manual for instructions. The most common symptom of failing o-rings is a failure of the tube leak-check procedure on the ATD. Dirty filters are indicated by contamination in blanks or system carryover. Also, periodic replacement of the multi-sorbent packing in the cold trap is required. After repacking the trap it should be baked out (Cold Trap Bake mode) at 20°C above the inject temperature until the CO_2 peak is minimized in system blanks and all contamination peaks are gone. Pre-packed cold traps are available from Perkin-Elmer and Markes.

On the Turbomatrix, the cold trap must be installed using PTFE ferrules instead of graphite or graphite/vespel ferrules. The latter two materials will allow significant air leaks into the GC. The PTFE ferrule must be retightened or replaced anytime the desorber is turned off and the heaters are allowed to cool. The cold trap is fragile and must be handled carefully to avoid breaking. When removing it, it is helpful to first heat the Swagelok fitting gently with a heat gun. This softens the ferrule and allows the trap to be removed without much force. On the Unity Series 2 and TD100, the seal is made with an o-ring. The o-ring only needs to be replaced when a leak is indicated by the leak check procedure. There are pictures and a short video in the Unity online help that show trap replacement.

5.3 GC System Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 9.4). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.4 Mass Spectrometer The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the backstreaming trap.

5.5 Instrument Tuning The instrument is tuned with guidance from the procedure described in the HP Operations Manual, when necessary. The tune shall meet the tune criteria described in this document.

6) Interferences

6.1 Sorbent Artifacts Typical artifact levels for ¼ inch O.D. tubes of 3.5" length range from 0.01ng and 0.1ng for carbonaceous sorbent and Tenax respectively. An exception is the presence of benzene from graphitized carbon blacks and carbon molecular sieves,

which may be difficult to reduce below 0.5ng per tube. Artifact levels are around 10ng for Chromosorb Century series and other porous polymer sorbents. However, these types of sorbent can still be used for low ppb monitoring if MS detectors are used or if the blank profile of the tube demonstrates that none of the sorbent artifacts interfere analytically with the compounds of interest.

The long-term storage of blank tubes will show levels of artifacts (0.01ng after 1 to 2 months and 0.1ng after 6 months) such as: Carbotrap/pack C, Carbotrap/pack B, Carbosieve SIII multi-bed tubes, and Tenax GR tubes. Artifact levels for porous polymers are higher – for example 5ng for Chromosorb 106 after 1 week.

Benzaldehyde, phenol and acetophenone artifacts are reported to be formed via oxidation of the polymer Tenax when sampling high concentration (100-500ppb) ozone atmospheres.

Stringent tube conditioning and careful tube capping and storage procedures are essential for minimizing artifacts. System and sorbent tube conditioning must be carried out using more stringent conditions of temperature, gas flow and time than those required for sample analysis. A reasonable objective is to reduce artifacts to 10% or less of individual analyte masses retained during sampling.

6.2 Water The capacity of the analytical instrumentation to accommodate the amount of water vapor collected on tubes is usually the limitation in obtaining successful results, particularly for GC/MSD applications. There are several approaches to reducing water interference during air monitoring using sorbent tubes. The first is to minimize water collection by selecting, where possible, a hydrophobic sorbent for the sample tube. This is possible for compounds ranging in volatility from n-C5. Tenax, Carbopack or one of the other hydrophobic sorbents listed in Method TO-17 (Table 2) should be used. The sampling volumes may also need to be reduced when the relative humidity is above 90%. The thermal desorber should allow for splitting of the sample before and/or after refocusing. Dry purging the sample tube with inert, dry gas prior to desorption is perhaps the most common water management tool. The desorber allows for a variable dry purge time and flowrate before thermal desorption. The tube may be heated while dry purging if the elevated temperature will not cause breakthrough of the target analytes or internal standards. A cold trap packed with multiple sorbents may be used which allows trapping of very volatile compounds at ambient temperatures while allowing most of the water to pass through.

6.3 Analytical System The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory tube blanks under the same conditions as sample tubes.

6.4 Carbon Dioxide Excessive levels of carbon dioxide present in a sample may interfere with analysis by freezing up the refocusing trap during initial desorption if using subambient cold trap temperatures. The CO_2 can usually be removed sufficiently by dry purging the sample tube prior to desorption. Ambient levels of CO_2 in sample air will not cause problems.

6.5 Glassware Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware which results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning*. The use of high purity water, reagents, and solvents helps to minimize these problems.

6.6 Particulates It may be necessary to connect a particulate filter (a 2 micron Teflon filter or short clean tube containing a loose plug of clean glass wool) to the sampling end of the tube in areas of extremely high particulate concentrations. Some compounds of interest may, however, be trapped on the Teflon or on the glass wool. Particulates trapped on the sorbent tube have the potential to act as a source or sink for volatiles, and may remain on the tube through several cycles of sampling and desorption. Replacement of the particulate filter is therefore recommended after each usage.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP may perform analysis, interpretation and peer review of the results.

7.2 The supervisor/manager must ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, matrix or test method are made.

7.3 The department supervisor/manager or designee shall perform final review and sign-off of the data.

7.4 All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. The requirements for performing these demonstrations in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual 5.0 are provided in the *SOP for Training Policy* and must be followed. The training plan (Attachment 1) shall be used to document the training certification of new analysts.

8) Sample Collection, Handling, and Preservation

8.1 New sampling tubes, even preconditioned purchased tubes, must be thermally conditioned and checked prior to deployment to the field. The most common sorbents and their maximum and suggested conditioning temperatures and flows are listed in Table 2 of Method TO-17. Conditioned tubes must be batch tested (a minimum of one in ten) using the parameters to be used for sample analysis. Tubes must be free of target analytes below the desired reporting limit.

8.2 Following the tube conditioning procedure, tubes must be sealed with Swagelok-type fittings and PTFE ferrules and wrapped and placed in an airtight container. Additionally, tubes shall be kept in a refrigerator (organic solvent-free) at <4ºC if not to be used within a day (method blank tubes or tubes for field use) and must be shipped to the field in a cooler packed with blue ice.

8.3 Field samples should be refrigerated at <4ºC in a clean environment during storage and analyzed within 30 days of sample collection (within one week for limonene, carene, bis-chloromethyl ether and labile sulfur or nitrogen-containing volatiles). Samples taken on tubes containing multiple sorbent beds should be analyzed as soon as possible (within two weeks) after sampling unless it is known in advance that storage will not cause significant sample recovery errors.

9) Equipment and Supplies

9.1 Gas Chromatograph (GC) An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

- Agilent 6890 Series

- Agilent 7890 Series

9.2 Thermal Desorber An instrument capable of two-stage thermal desorption, where the sample tube is thermally desorbed onto a refocusing trap, then rapidly transferred to the GC analytical column. The analytes should be desorbed from the sample tube and refocusing trap in backflush mode, i.e. in the opposite direction of flow during sampling.

The desorber should be capable of leak checking the sample tube and flow path, and of splitting the sample flow both before and after refocusing. It should also be equipped with a gas-loop internal standard addition option that loads an aliquot of the gas-phase internal standard onto the front of the sample tube before desorption.

- Perkin-Elmer TurboMatrix Automated Thermal Desorber
- Markes Unity/Ultra Series 2 Automated Thermal Desorber
- Markes TD100 Automated Thermal Desorber

9.3 Mass Spectrometer (MS) A MS capable of scanning from 33 to 300 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

- Agilent 5973 Network Mass Selective Detector or equivalent
- Agilent 5975 Mass Selective Detector or equivalent

9.3.1 Ionization Gauge Controller

Granville-Phillips 330 Ionization Gauge Controller: 330001/2/3
Hewlett Packard Ionization Gauge Controller: 59864B

9.4 Analytical Column Any analytical column capable of separating the compounds of interest may be used. The capillary column should be directly coupled to the source of the mass spectrometer. The following are suggested columns; an alternative column may be used as long as sufficient peak resolution and separation is achieved.

Column: Phenomenex ZB-1MS Fused Silica Capillary Column
60m x 0.25mm ID
1.0 micron film thickness

OR

Restek Rxi-1ms Fused Silica Capillary Column
30m x 0.25mm ID
1.0 micron film thickness

9.5 Data Systems IBM-compatible PC with Windows 95/98/NT/XP/Vista/7 and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library or equivalent.

9.6 Tube Spiking Apparatus A device that allows a sorbent tube to be connected to a heated inlet with a septum port and a flow of inert gas. A GC injection port with a glass or fused silica-lined insert works well. A ¼" teflon union with teflon ferrules is used to connect the tube to the port, and a flow of high purity helium or nitrogen is set at 50 – 100 ml/min. The inlet is heated to 50-200°C depending on the boiling points of the analytes being spiked. Alternatively, for semi-volatile analytes, standard can be directly spiked onto the front of the tube at ambient temperature and purged with inert gas.

9.7 Sample Collection Tubes Perkin-Elmer type stainless steel, glass, or glass-lined stainless steel tubes, 3.5" x 0.25".

The tubes are packed with solid sorbent material, typically 60/80 mesh, held in place with stainless steel gauze and/or glass wool plugs. A steel retaining spring is used in the back of the stainless steel tubes. The front end (sampling inlet) is marked by one or two inscribed rings (stainless steel tubes) or a glass frit (glass tubes).

Each tube must be clearly marked with a unique serial number. Tube manufacturers typically etch the serial number on the outside of the tube.

The appropriate sorbent tube must be selected for the analytes being tested. There are many literature sources describing the properties of various packings and their suitability for trapping chemicals of different boiling points and functional groups. A tube containing multiple beds of sorbents of increasing strength (front to back) is the most versatile and is used by the laboratory for sampling and analysis of the compounds listed in Table 2. Either Carbotrap 300 or Perkin Elmer SVI tubes are acceptable since they contain equivalent packings. If the compounds of interest are less volatile than benzene, a tube with a single bed of Tenax TA may be used. Selection of tubes for non-routine compounds should be done with the assistance of the literature available from the manufacturers, such as Perkin-Elmer and Markes International.

9.8 Diffusion Caps Diffusion caps are needed when tubes are used for passive diffusion sampling. The caps are placed on the inlet end of the tube (while the back end remains sealed) and are held in place by a viton o-ring. The tube is then placed in a fixed location for as long as four weeks, during which VOCs are adsorbed into the sorbent bed by diffusion at a very slow rate. The caps are available with a silicone membrane that reduces water adsorption during the sampling period.

9.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent. Used for preparing standards.

9.10 Dynamic Dilution System

- Entech Dynamic Diluter Model 4620A
- Toshiba laptop computer Model 2210CDT/6.0 and Software NT460

10) Standards and Reagents

10.1 Reagents

10.1.1 UHP Grade Helium (99.999%)(GC carrier gas and tube-spiking sweep gas)

10.1.2 UHP/Zero Grade Air (TurboMatrix pneumatics gas)

10.1.3 Purge and Trap Grade Methanol

10.1.4 Cryogen - Liquid nitrogen from bulk tank (used to cool preconcentrator traps)

10.2 Standards Calibration and QC standards are prepared by spiking known amounts of target analytes onto pre-cleaned tubes of the same sorbent as the sample tubes. Standards may be gas-phase or stocks prepared in solvents (usually methanol).

10.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Standard Prepare a standard mixture containing three system monitoring compounds (1,2-Dichloroethane-d4, Toluene-d8 and p-Bromofluorobenzene (also used as a tune check)) and three internal standards (Bromochloromethane, Chlorobenzene-d5 and 1,4-Difluorobenzene) at 50 µg/L each in humidified zero air in a Summa canister. This canister is connected to the thermal desorber Internal Standard Option inlet using a small regulator to reduce the

canister outlet pressure to 10psig. Other concentrations may be used depending on the application and the size of the sample loop.

10.2.1.1 An intermediate standard is prepared from neat compounds in a small glass vial by combining equal mass amounts of each neat compound using a microliter syringe.

Step 1: This cocktail is prepared by combining 25mg (25,000 µg) of each neat compound. Take the density of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$V_s = \frac{A}{d} \qquad \text{(Equation 1)}$$

Where:

V_s Actual spike amount (µl)
A Desired amount for each compound (µg)
d Density (µg/µl); refer to Table 3 for the density

Example: Calculate the actual amount of p-bromofluorobenzene to add to the cocktail:

$$V_s = \frac{25000\mu g}{1543\mu g/\mu l} = 16.2\mu l$$

Step 2: The concentration of each compound in the cocktail is determined by the following equation.

$$C = \frac{A}{V} \qquad \text{(Equation 2)}$$

Where:

C Concentration of cocktail (µg/µl)
A Amount of each compound (µg)
V Final volume of cocktail (total spike amounts of each compd., µl)

Example:

$$C = \frac{50000\mu g}{144.2\mu l} = 346.7\mu g/\mu l$$

Step 3: Prepare the Summa canister standard by spiking a small amount of the neat mix directly into the can while filling with humid zero air. Use a tee with a septum on one port, spiking the neat mix in while the can is still under vacuum, then fill to 83.3 psig. This gives a final volume in the can of 40 liters. Calculate the spike volume using the following equation:

$$V_s = \frac{V_c C}{d}$$ (Equation 3)

Where:

V_s Spike volume (µl)
V_c Final canister volume (L)
C Desired canister conc. (µg/L)
d Density of each compound in neat mix (µg/µl)

Example: For a 50 µg/L standard in a six-liter Summa canister at 83.3 psig:

$$Vs = \frac{(50\mu g/L)(40L)}{(346.7\mu g/\mu l)} = 5.77\mu l$$

Density (µg/µl)	Compound
1934.4	Bromochloromethane
1307	1,2-Dichloroethane-d4
1170.1	1,4-Difluorobenzene
943	Toluene-d8
1157	Chlorobenzene-d5
1543	p-Bromofluorobenzene

The final volume of the Summa canister is calculated using the following equation:

$$V_f = (PDF)(V_i) = \frac{(P_{atm} + P_f)}{(P_{atm+P_i})}(V_i)$$ (Equation 4)

Where:

V_f Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PDF = $\frac{P_{atm} + P_f}{P_{atm} + P_i}$

P_f Final Canister Pressure (psig)

P_i Initial Canister Pressure before dilution (1 atm, in psig)

V_i Volume of canister at 1atm (L)

Example:

$$V_f = \frac{(14.7 + 83.3)}{14.7 + 0}(6.0L) = 40L$$

10.2.2 Initial Calibration (ICAL) Standards Calibration standard stocks for spiking tubes may be prepared in several ways, either in the gas phase (static dilution bottles, Summa canisters, certified cylinders) or in solvents (methanol or water stocks).

The choice depends on the analytes of interest and the sorbent being used for sampling. If methanol stocks are used, it is necessary to prepare them at concentrations that allow for a very small spiking volume, usually less than ten microliters. Strong adsorbents such as carbon molecular sieves may require spiking volumes of one microliter or less if no inlet split is used during desorption. Standards prepared in Summa canisters are prepared according to the procedure detailed the VOA-TO15 SOP.

10.2.2.1 A gas-phase stock standard is prepared from neat compounds in a glass static dilution bottle (SDB). It may be more practical to prepare equi-mass mixes (compounds in equal mass amounts) from the neat compounds before preparing the SDB.

Step 1: A neat mix is prepared by combining 25mg (25,000µg) or more of each neat compound in a glass vial with a teflon-lined cap. Calculate the microliter volume of each compound needed using Equation 1.

Example: Calculate the actual volume of benzene to add to a cocktail containing 50000µg of each compound:

$$V_s = \frac{50000\mu g}{876.5\mu g/\mu l} = 57.0\mu l$$

Step 2: The concentration of each compound in the cocktail is determined using Equation 2.

10.2.2.2 A gas-phase standard is prepared by spiking an aliquot of a neat cocktail into an SDB. The SDB is then heated from a minimum of one hour at 60°C to completely volatilize all components. The spike amount is determined by using the following equation.

$$V_S = \frac{V_{SDB} C_{SDB}}{C_S} \qquad \text{(Equation 5)}$$

Where:

V_S Spike volume required (µl)
C_{SDB} Desired concentration (µg/ml)
C_S Concentration of compound in cocktail (µg/µl)
V_{SDB} Actual volume of SDB (ml)

10.2.2.3 Working Standards are prepared by spiking an aliquot of standard stock onto a clean sorbent tube while flushing with clean diluent gas at 50-100 ml/min.

Calculate the spike volume needed using the following equation:

$$V_S = \frac{A}{C_{SDB}} \qquad \text{(Equation 6)}$$

Where:

V_S Spike Volume needed (ml)
A Desired Mass Amount (µg)
C_{SDB} Conc. of SDB (µg/ml)

10.2.3 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) Prepare a second source standard (either a different manufacturer or lot from the same manufacturer as the initial calibration standard) with all the analytes used in the calibration standard.

10.2.4 Continuing Calibration Verification Standard (CCV) The CCV is the same standard used for the initial calibration.

10.3 Storage and Expiration Dates

- Neat Stock Liquids – are stored at -10˚C to -20˚C for a period of three years or as specified by the manufacturer.
- Equi-Mass Primary Stock Standard – is a cocktail or soup of neat compounds (containing compounds in equal mass amounts) used in preparing intermediate gas phase standards and shall be stored in the freezer at -10˚C to -20˚C for up to six months. This is assuming that the soup is sealed with a septum-containing screw cap or Mininert™ valve. The selection of the compounds for the soup should be performed in accordance with the guidelines in Volume 6.5 of the *Tekmar-Dohrmann* Application Note.
- Stock Standards purchased in cylinders must be stored at laboratory temperature for a period of 2 years or as specified by the manufacturer.
- Intermediate Calibration Standards prepared by static dilution may be stored at room temperature, but must be heated in an oven at approximately 60°C for at least one hour before use to ensure analyte vaporization. To increase the useful lifetime of an SDB standard, remove volumes of 25ml or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, an SDB intermediate standard is stable for approximately two months as long as new working standards made from this standard continue to meet acceptance criteria. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar-Dohrmann* Application Note.
- Calibration Standards prepared in canisters may be stored at laboratory conditions for thirty days in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

11) Method Calibration

11.1 Initial Calibration Refer to Section 16.4 for the technical requirements for performing an initial calibration.

11.1.1 Calibration Points Analyze a minimum of five levels of the calibration standard (analyze low to high) that span the monitoring range of interest of the samples. The range is typically within 0.5ng-100ng per tube. The dynamic range is dependent on the sensitivity of the particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly.

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds. One of the

calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard. Also, one of the standards must be at or below the method reporting limit for the compounds of interest or the MRL must be adjusted accordingly.

Following initial calibration standard analysis, an ICV standard must run within 24 hours of the initial calibration BFB tune. The concentration of the ICV standard should be at or below the midpoint of the calibration range.

11.1.2 Recalibration The GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument or if the continuing calibration verification acceptance criteria have not been met as specified in Section 16.6.

11.1.3 Analytical Window If time remains in the 24-hour tune window after meeting the acceptance criteria for the initial calibration and ICV, samples may be analyzed according to the procedure described in this document (see Section 12.2). If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.1.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 12.3. Use the standard prepared in accordance with Section 10.2.2 of this SOP.

Use the same tube media used for field sampling. Prepare the calibration tubes by spiking with the appropriate volume of calibration stock standard for each level. For standards prepared in solvents, use spiking stocks made at a concentration that allows for a spiking volume of 0.1µl to 10µl. Volumes of less than 1.0µl are best because they introduce less solvent onto the tube. Connect the tube to the bottom of the spiking rig (usually a GC injector port) using a PTFE ferrule (finger tight). The injector may be heated up to 200C depending on the volatility of the target analytes. Set the gas flow to 50 to 100 ml/min and spike the standard through the septum port. Allow the spike to be swept completely onto the adsorbent bed of the tube and remove most of the solvent before removing from the injector port (approximately two minutes). Place analytical caps on the tubes and put them in order from low to high concentration on the autosampler and analyze using the same parameters that will be used for samples.

For gas-phase standards prepared in Summa canisters or Tedlar bags, the standard can be pulled onto the tube using a ground-glass syringe. Typical volumes used are from 5ml to 500ml. Connect the inlet side of the tube to the canister valve using a PTFE ferrule (finger tight) and then connect the syringe to the outlet end using silicone tubing that makes a leak-tight seal. Open the canister valve very slowly until the syringe plunger starts to move. The ideal rate is 50 to 100 ml/min so use the valve to regulate the flow rate. Close the valve when the desired volume is reached. Place analytical caps on the tubes and put them in order from low to high concentration on the autosampler and analyze using the same parameters that will be used for samples.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 2 and the primary ion should be used unless interferences are present, in which case the secondary ion may be used. Refer to Section 15.1 for the required calculations and Section 16.4.2 for the acceptance criteria.

11.1.5 ICAL Method Update Procedure Initial calibration methods are updated using the following procedure.

1. Open the current ICAL method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID is the date files were analyzed.
3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Re-quant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not re-quant datafiles.
7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.
12. Upon completion of ICAL work-up, quantitate the ICV and verify results with acceptance criteria (see Section 16.5).

11.1.6 Initial Calibration Review Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment 2 must be used to document the review and approval process.

Analyte concentrations, which are not "real", not to be reported, or otherwise marked off the initial calibration, should be followed by a short explanation regarding the reason for the omission.

11.1.7 Initial Calibration File An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Blank analysis quantitation report
- Calibration status report (aka Calibration History)

- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and %recovery report
- Calibration standard and secondary source concentrations
- Copy of instrument run log

11.2 Initial Calibration Verification Standard Verify the initial calibration by analyzing an initial calibration verification standard (ICV) within 24 hours of the BFB tune. This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 10.2.3.

Inject an aliquot of the ICV standard onto the same type of tube used for the ICAL and analyze under the same conditions. Refer to Section 15.3 for the required calculations and Section 16.5 for the acceptance criteria.

11.3 LOQ Establishment, Verification, and Acceptance Criteria

1. The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
2. The LOQ for each analyte must be ≥ the analyte's LOD.
3. Initially a passing demonstration of precision and bias must be performed at the LOQ.
4. Run CCV 2 times at LOQ and:
   a. Generate a duplicate report for precision using ±25% as the criteria.
   b. Check the %Rec using current laboratory control limits.
   c. Check the signal to noise ratio (S/N) using the software. The S/N ratio must be at least 3:1 for each analyte.
   d. All ion abundances must be acceptable per the requirements set forth in this document.
5. If any compounds fail, verify at a higher level and notify reporting. Also, make a note in the ICAL documentation.
6. Turn in all LOQ verification data (quant reports and software reports/checks) to QA (regardless of pass/fail).
7. Verify the LOQ on each instrument quarterly for DoD compliance.

12) Sample Preparation/Analysis

12.1 Sample Preparation

12.1.1 Sample Tubes Sample tubes must be removed from cold storage and allowed to warm up to room temperature prior to analysis. Remove the Swagelok-type storage caps and replace with analytical caps before loading tubes onto autosampler. Check for sample integrity, i.e. loose retaining gauze, loose sorbent pieces at front and back, writing/markings on tube, deformation of tube ends from over tightening caps, or particulate on inlet gauze. Clean as necessary (without solvents), but be careful not to dislodge retaining gauze. Note any sample integrity findings on the *Sample Preparation and Analysis Observations* (yellow) sheet and notify the project managers about any anomaly that may compromise the analysis.

The serial number of each tube analyzed (samples, standards, and blanks) must be recorded in the header information of the datafiles so that it will appear in the sample run log.

12.2 Analytical Sequence For this internal standard calibration method analysis, a CCV standard is to be analyzed every 24 hours. That is, the last analysis in the sequence must be started within 24 hours from the time of the initiation of the sequence. The initiation is considered to be the injection of the BFB tune standard.

The analytical sequence must be completed for the analysis of ≤20 field samples. A tube blank (MB) shall be run to monitor for system contamination. There must be at a minimum a laboratory control sample and duplicate (LCS/LCSD) analyzed in each batch to assess batch precision. The concentration of the LCS (ICV standard) should be at the lower end of the calibration curve as an indication that the system allows for good recovery at those concentrations. The following generalized analytical sequence is to be followed:

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Levels Minimum) ICV Standard[2] (Acts as the ICV and LCS) Tube Blank LCSD Standard Up to 20 Field Samples
With Continuing Calibration	Tune Check[1] CCV Standard[4] Tube Blank LCS/LCSD[3] Up to 20 Field Samples

[1] The introduction of the tune check standard is the start of the 24 hour analysis window. The instrument performance check solution must be analyzed initially and once per 24 hour time period of operation. The BFB from the CCV run or the first calibration standard run may be used for this purpose.
[2] In this scenario, the ICV may also be evaluated as the LCS, as long as an LCSD is also analyzed.
[3] An LCS/LCSD shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard analyzed at the lower end of the calibration curve.
[4] A CCV must be analyzed at the beginning of every analytical sequence.

12.3 Conditions

12.3.1 Thermal Desorber Conditions The suggested settings and system parameters for Tenax and multi-bed tubes are as follows:

Perkin Elmer TurboMatrix ATD:

Desorb	Tenax TA	Carbopack/Carbosieve
Transfer Line:	200˚C	200˚C
Valve Temp:	200˚C	200˚C
Dry Purge:	1-5 min	5-20 min
Dry Purge Flow:	50-100 ml/min	50-100 ml/min
Desorb Temp.:	250˚C	300˚C to 350˚C
Desorb Flow Rate:	25-100 ml/min	25-100 ml/min
Desorb Time:	5-10 min	10 to 15 min

Inlet Split:	As needed	As needed

Refocusing Trap

Temperature:	-10°C to 25°C (depending on trap packing)
Injection Temp.:	290°C
Injection Time:	3.0 min
Outlet Split	3:1 minimum (split flow:column flow)

Markes Unity/Ultra Series 2 and TD100:

Desorb	Tenax TA	Carbopack/Carbosieve
Transfer Line:	140°C	140°C
Valve Temp:	140°C	140°C
Dry Purge:	1-5 min	5-20 min
Dry Purge Flow:	50-100 ml/min	50-100 ml/min
Desorb Temp.:	250°C	300°C to 350°C
Desorb Flow Rate:	25-100 ml/min	25-100 ml/min
Desorb Time:	5-10 min	10 to 15 min
Inlet Split:	As needed	As needed

Refocusing Trap

Temperature:	20°C (depending on trap packing)
Injection Temp.:	300°C
Injection Time:	3.0 min
Outlet Split	Can be run splitless; typically 5:1 minimum

Adsorbent Tube Reconditioning Conditions

Temperature:	at least 20°C above sample desorb temperature
Initial Bakeout:	2 hours or until clean blank is obtained

12.3.2 GC/MS System Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.5mL/min (head pressure ~ 17 psi)
Temperature Program	Initial Temperature: 40°C Initial Hold Temperature: 3 min Ramp Rate: 5°C/min to 125°C 2nd Ramp: 20°C/min to 240°C for 6 min hold
Detector B (MSD *Interface)*:	270°C
Electron Energy	70 Volts (nominal)
Mass Range	33 to 300 amu (SCAN mode)
Mass Range	Scan masses corresponding to the target analytes (SIM mode)
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

Note: The instrument may be operated in Selective Ion Monitoring (SIM) mode if requested by the client.

12.4 Instrument Performance Check Since the BFB tuning compound is included in the internal standard and surrogate standard canister and an autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The following is the procedure to follow when performing the instrument performance check.

- Inject 50ng BFB or less (on column). A minimum of 1 to 5ng is typically necessary to achieve a passing tune evaluation depending on the instrument. The IS canister is at 50µg/L. The IS loop on the Perkin Elmer TurboMatrix is 0.5 ml, which gives a 25 ng spike onto each tube. The IS loop on the Markes Unity/Ultra Series 2 and TD100 is 1.0 ml which gives a 50 ng spike onto each tube. The amount on column is reduced by the split ratios set up in the method. It may be necessary to analyze the BFB tuning check with a lower overall split ratio than the samples to get adequate mass on column.
- The BFB must be checked using the automated tune evaluation routine in the GC/MS data analysis software. This takes a three-scan average spectrum about the apex and performs a single-scan background subtraction. This evaluation must meet the method criteria before samples can be analyzed. Any QC (ICAL or daily QC runs) analyzed after a failed tune evaluation must be reanalyzed.

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions. Refer to Table 1 for the acceptance criteria and Section 16.3 for the required corrective action.

12.5 Continuing Calibration Verification Standard Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the 24-hour tune window has closed) by analyzing a continuing calibration verification (CCV) standard prepared from the calibration standard stock, in the same manner as the ICAL standards. While it is suggested that the concentration of the calibration verification be near the midpoint, it is acceptable to vary the concentration within the established calibration range.

Refer to Section 15.2 for the required calculations and Section 16.6 for the acceptance criteria.

12.6 Method Blank A clean tube of the type used for calibration and samples must be analyzed after the CCV standard to demonstrate that the system is free of contaminants that would interfere with sample analysis. Place a pre-cleaned tube on the autosampler and analyze using the same parameters used for standards and samples. If stored cold, allow the tube to warm to room temperature first.

12.7 Laboratory Control Sample and Duplicate (LCS/LCSD) The laboratory control sample is an analysis of the initial calibration verification standard. Inject the LCS (ICV) onto a pre-cleaned tube (same media type as for ICAL and samples) at a concentration equal to or below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst. Since it is not possible to analyze a

true laboratory sample duplicate, a duplicate LCS should be analyzed with each batch to assess method precision.

12.8 Sample Analysis

12.8.1 Procedure Prior to analysis, all sample tubes should be at temperature equilibrium with the laboratory. After achieving acceptable results for the tuning check, CCV standard, blank, and LCS, load the sample tubes onto the autosampler carousel and set up the method and sequence. Use the same method as used for all standards and blanks.

Following completion of the analytical sequence the sample chromatograms and instrument sequence report must be checked prior to removing tubes for reconditioning to ensure all samples were analyzed correctly. If any anomalies are identified the tubes should be recapped so that reanalysis can occur if necessary. The sequence reports are saved to the computer drive where the control software resides.

Check all target compounds using the QEdit routine in Enviroquant, making sure all extracted ion chromatogram peaks are integrated properly.

Note: The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).

Refer to Section 15.5 for the required calculations and check the internal standard peak areas and retention times as well as applicable surrogate recoveries to see if they meet acceptance criteria (see Section 16.9).

SCAN Mode

The instrument is normally operated in the SCAN mode, where the following procedure may be followed.

- Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic range from 33 to 300 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra. See operating conditions in Section 12.3.
- If reporting Tentatively Identified Compounds (TICs), refer to Section 12.8.2 for identification criteria.

SIM Mode

When the client requests SIM mode, select SIM instead of SCAN mode and identify a minimum of two ions per analyte of interest. Also, a minimum of two ions for each internal standard and surrogate compound should be selected.

12.8.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

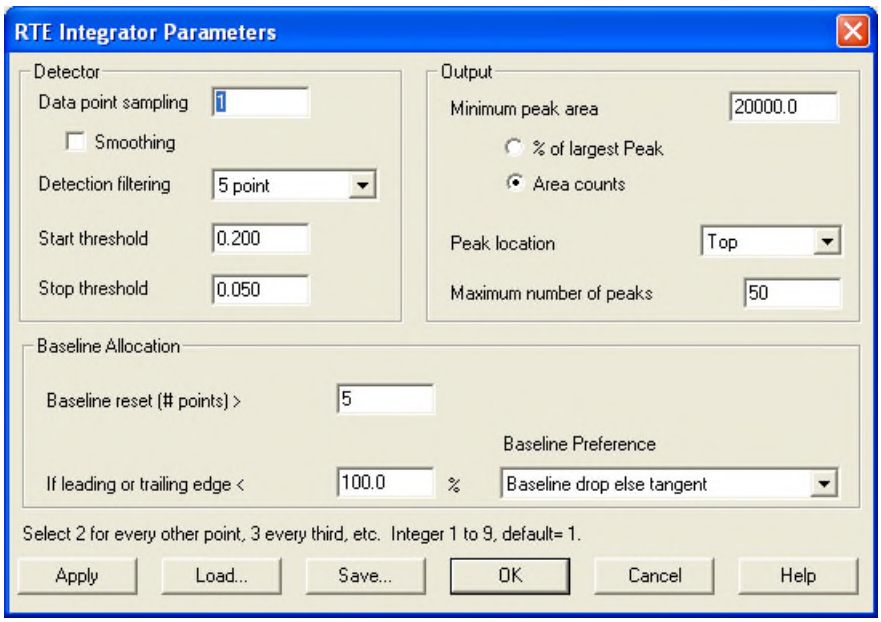
Certain programs may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the

analyst assign a tentative identification. The following guidelines are used for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. For example, for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30 and 70%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- The concentration of the tentatively identified compound is estimated by assuming a response factor of 1.0 and comparing the response of the tentatively identified compound to the response of the closest internal standard. If internal standard co-elution occurred, the next closest internal standard will be used for calculation of the tentatively identified compound concentration. If all internal standards in the sample display co-elution, then an external standard quantitation method should be used. The internal standard used for quantitation will be the second internal standard in the method blank.

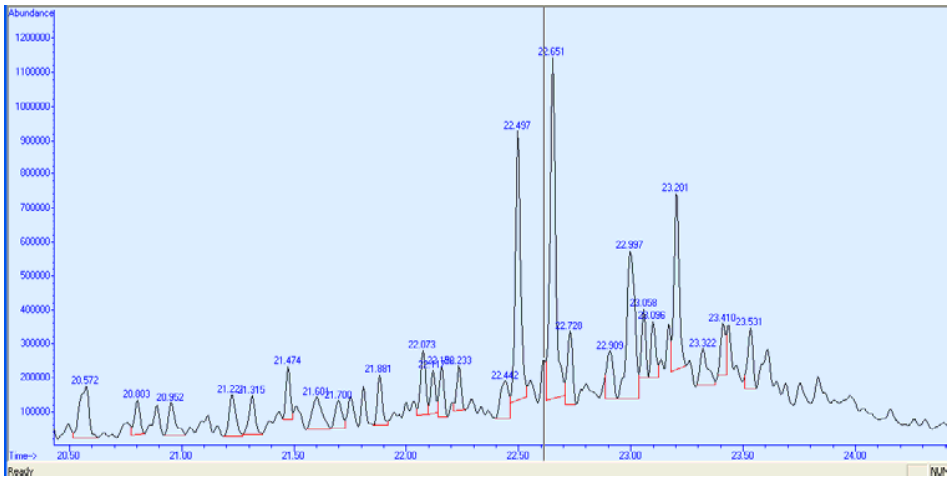
<u>Procedure for Reporting Tentatively Identified Compounds (TICs)</u>

1. Load the datafile in the main Enviroquant window.
2. Load the TIC integration parameters (LSCINT.p). Typical setpoints are as shown below.

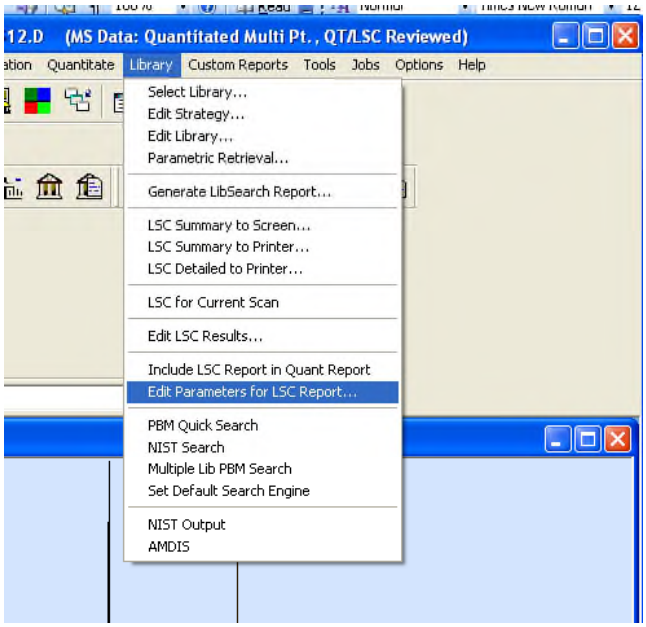


3. Integrate the chromatogram and inspect the peak integrations. Adjust the parameters as needed to achieve integration that will:

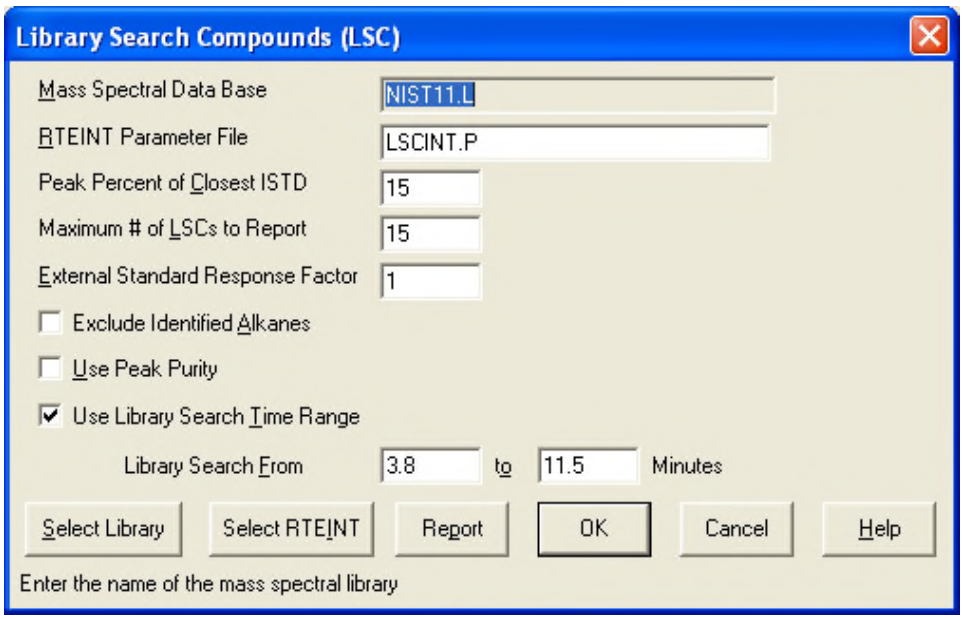
   - Resolve closely-eluting peaks that only have a small valley separating them.
   - Not include excess area below the peak in a complex matrix with an elevated baseline.
   - Include peak tailing when necessary.
   - Yield a sufficient number of peaks that will ensure that the internal standards are included.



4. Edit the parameters to be used in generation the library search report:



Select the most current mass spectral library database available, the correct integration parameters file, the area threshold (as a percent of IS area), number of peaks to report, and a time range of the chromatogram to search (set to start after the CO2 peak).



5. Run the LSC routine from the Library menu. You may choose 'LSC Summary to Screen' (Calculate/Generate Report) to get a quick view of the results and then proceed if they seem acceptable. Set the default printer to 'Adobe PDF' and then choose 'LSC Detailed to Printer'.
6. Open the pdf file and inspect the LSC summary (last page). Check the internal standard areas and confirm that they are correct. If any IS area is biased high due to a coeluting peak use the 'Edit LSC Results' routine to switch all associated TICs to use a different IS. If all three IS peaks have coelutions substitute the areas from the daily method blank in the calculations.
7. Use the LSC Summary as a guide and inspect the chromatogram in the data analysis window. Integrate the chromatogram from the Integrate menu and look for peaks that may have been missed by the LSC routine. Possible reasons for missed peaks are excessive tailing (organic acids), RT close to a target compound, coeluting peaks with no valley between them. These will need to be added manually.
8. Use the DOSCAN routine from the Tools menu to search individual missed peaks one by one. This will add them to the LSC list.
9. Go back into the Edit LSC Results routine and make any necessary changes to compound names and/or the internal standard used for quantitation.
10. Run the macro "QT '0,0,C' by clicking the Custom Tool 1 button. This will update the LSC list to the quant.csv file.
11. Run the LSC Detailed to Printer routine from the Library menu (Generate Report *only*). This will print the file to pdf.
12. Excel Reporting
    1. In Excel, open the TIC reporting template (I:\A-GCMS\TICS\System\StarLIMS_TICQ).
    2. Enter the service request number and click ok.
    3. Click the Get Samples button. Select the samples to be reported. Delete any samples that are not to be reported (right click/delete row).
    4. Click the Update PEF button.

5. Click the Get TICs from CSV button. Enter the date analyzed and select the instrument ID.
6. Click the Apply to all Samples button. Change the date for any sample that was analyzed on a different date.
7. Click the Apply Instrument to all Samples button.
8. Enter file number in column E (i.e. enter 07 for file 12301507.d).
9. Click the Copy Data button. This copies the TIC info to the report sheets.

12.9 Manual Integration The integration for each peak shall be checked to ensure that it has been integrated properly. Assuming an incorrect automatic integration the analyst shall conduct the manual integration in accordance with the *SOP for Manual Integration Policy* including all documentation and reviews associated with the process. The review shall include the analyst and reviewer initialing and dating the manual integration as an indication of acceptability and approval.

Reporting Requirements Certain project requirements including samples which are submitted under the Department of Defense (DoD) QSM require that the case narrative include an identification of samples and analytes for which manual integration is required. Refer to project requirements to determine if this is necessary.

12.10 Method Detection Limits and Limits of Detection A method detection limit study or MDL/LOD verification shall be performed annually on each instrument for which this method is performed. The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The higher detection limit (from all of the instruments where this method is to be performed) for each analyte must be selected and used as the uniform method detection limit for that analyte regardless of the instrument of analysis. The detection limit shall be used to determine the LOD for each analyte.

Once determined on each instrument, the LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD.

12.10.1 Performance and Acceptance Criteria

1. The MDL must be <0.5ppbV for each analyte (Method 14.1).
2. Perform Limit of Detection (LOD) verification on all instruments (performing this method) immediately following the MDL study. Spike the LOD at 2-4x the MDL; the spike level establishes the LOD.
3. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - For DoD compliance the LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
4. If the LOD verification fails (per #3), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
5. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Data System Setup

For the HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Operating and acquisition parameters will change depending on the target analytes. Typical GC/MS operating parameters for VOCs are given in Section 12.3.2.

For the Agilent systems with the Markes desorber, consult the online help or user manual for basic operation instructions. Choose the appropriate desorption method for the tube type and analytes, set up the sequence, and load the tubes into the autosampler trays. The tube IDs and tray positions must be double checked against the service request form and data systems sequence before loading trays into the TD autosampler. Then set up the GC/MS acquisition parameters. The desorber will not inject a sample unless the GC/MS is in ready mode.

14.2 Storing Electronic Data The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. There are multiple quantitation methods, which are subsets of the compound list in Table 2. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

14.3 Sufficient raw data records must be retained of the analysis, instrument calibrations and method detection limit studies including: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. Retain all daily QC per sequence on file for future reference including tune checks, opening standards, method blanks, laboratory control samples, laboratory duplicates, and initial calibrations and initial calibration verifications. Additionally, all passing QC tube checks must also be retained on file.

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

14.4 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation, standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 Initial Calibration

- Calculate the RRF for each target compound relative to the appropriate internal standard (refer to Table 2) using equation number 1 in Section 15.7.1.
- Calculate the mean RRF (using equation number 2) for each compound by averaging the values obtained from each concentration.
- Using RRFs from the initial calibration, calculate the %RSD for all target compounds using the equations 3 and 4 in Sections 15.7.3 and 15.7.4.
- Calculate the RRT for each compound over the initial calibration range using equation number 9 in Section 15.7.9.
- Calculate the mean RRT for each analyte target compound over the initial calibration range using equation number 10 in Section 15.7.10.
- Tabulate the area response (Y) of the primary ions listed in Table 2 and the corresponding concentration for each compound and internal standard.
- Calculate the mean area response $\overline{Y}$ for each internal standard compound over the initial calibration range using equation number 11 in Section 15.7.11.
- Calculate the mean of the retention times for each internal standard over the initial calibration range using equation number 12 in Section 15.7.12.

15.2 Continuing Calibration Verification

- The area response of the primary quantitation ion is used unless interference is observed. Calculate the relative response factor (RRF) of each target compound using equation number 1.
- Calculate the percent difference (%D) in the RRF of the CCV compared to the mean RRF in the most recent initial calibration using equation number 5 in Section 15.7.5.

15.3 Initial Calibration Verification and Laboratory Control Sample

- Calculate the percent recovery using equation number 13 (15.7.13).

15.4 Duplicate Analysis

- The relative percent difference of the LCS/LCSD must be calculated using equation number 14 (15.7.14).

15.5 Sample Analysis

- Calculate the retention time difference.
- Calculate surrogate recoveries using equation number 13.
- Calculate the change in area response using equation number 14.
- Calculate analyte concentrations (ng on column) using equation number 6 in Section 15.7.6.
- Calculate the final sample concentration using equation numbers 7 and 8 in Sections 15.7.7 and 15.7.8.

15.6 Method Blank

- Calculate the retention time difference
- Calculate the change in area response using equation number 14.
- Calculate analyte concentrations (ng on column) using equation number 6 in Section 15.7.6.
- Calculate the final sample concentration using equation numbers 7 and 8 in Sections 15.7.7 and 15.7.8.

15.7 Calculations

15.7.1 Equation Number 1

Relative Response Factor (RRF):

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where:

A_x	is the area response of the analyte quantitation ion.
A_{is}	is the area response of the corresponding internal standard quantitation ion.
C_{is}	Internal standard concentration, ng.
C_x	Analyte concentration, ng.

Note: The equation above is valid under the condition that the volume and concentration of internal standard spiking mixture added in all field and QC samples is the same from run to run.

15.7.2 Equation Number 2

Average (or Mean) RRF:

$$\overline{RRF} = \frac{\sum_{i=1}^{N} RRF_i}{N}$$

where:

RRF_i	are the individual RRFs from each concentration level in the initial calibration curve.
N	is the number of calibration concentration levels.

15.7.3 Equation Number 3

Standard Deviation, SD:

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(RRF_i - \overline{RRF}\right)^2}{N-1}}$$

where:

RRF_i	are the individual RRFs from each concentration level in the initial calibration curve.
$\overline{RRF}$	Average (or Mean) RRF of all concentration levels in the initial calibration curve.
N	total number of calibration concentration levels

15.7.4 Equation Number 4

Percent Relative Standard Deviation, %RSD:

$$\%RSD = \frac{SD}{\overline{RRF}}(100)$$

where:

SD Standard Deviation calculated in equation number 3
$\overline{RRF}$ Average or Mean RRF

15.7.5 Equation Number 5

Percent Difference, %D (CCV)
The %D is used for evaluating CCV RRFs vs. the initial calibration $\overline{RRF}$:

$$\%D = \frac{RRF_{CCV} - \overline{RRF}}{\overline{RRF}}(100)$$

where, for any given analyte:

RRF_{CCV} is the RRF from the CCV being evaluated.
$\overline{RRF}$ is the mean RRF from the current calibration curve.

15.7.6 Equation Number 6 For calculating analyte concentrations in a sample, the starting point is the nanogram amount generated by the HP Enviroquant software, which appears on the quantitation report. This will be the mass collected on the sampling tube (ng/tube). The equation used is:

$$ng_x = \frac{A_x ng_{is}}{A_{is}\overline{RRF}}$$

where:

ng_x is the nanogram amount of analyte *x*.
A_x is the area response of the analyte's quantitation ion.
A_{is} is the area response of the corresponding internal standard's quantitation ion.
ng_{is} is the internal standard amount, in nanograms.
$\overline{RRF}$ is the average or mean RRFs

15.7.7 Equation Number 7 The final analyte concentration, C_x, in units of micrograms per cubic meter (µg/m³), is then calculated from ng_x using the following:

$$C_x = \left(\frac{ng_x}{V}\right)\left(\frac{1\mu g}{1000ng}\right)\left(\frac{1000l}{1m^3}\right)$$

where:

V is the sample volume, in liters.

15.7.8 Equation Number 8 To convert to units of parts per billion volume (ppbv), the equation is:

$$C_{ppbv} = C_x\left(\frac{24.46}{FW}\right)$$

where:

FW is the formula weight of the analyte, in g/mol.
24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).

C_x the final analyte concentration in micrograms per cubic meter.

Refer to Table 2 for the appropriate molecular weights.

15.7.9 Equation Number 9

Relative Retention Time (RRT)

$$\mathrm{RRT} = \frac{\mathrm{RT_C}}{\mathrm{RT_{is}}}$$

where:

$\mathrm{RT_C}$ Retention time of the target compound, seconds.
$\mathrm{RT_{is}}$ Retention time of the internal standard, seconds.

15.7.10 Equation Number 10

Mean Relative Retention Time ($\overline{RRT}$)

$$\overline{RRT} = \sum_{i=1}^{n}\frac{RRT_i}{n}$$

where:

$\overline{RRT}$ Mean relative retention time for the target compound for all initial calibration levels.
$\mathrm{RRT_i}$ Relative retention time for the target compound in level i.
n Number of calibration levels

15.7.11 Equation Number 11

Mean Area Response ($\overline{Y}$) for Internal Standard

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$

where:

Y_i Area response for the primary quantitation ion for the internal standard for each initial calibration standard.
n number of calibration concentration levels

15.7.12 Equation Number 12

Mean Retention Times ($\overline{RT}$)

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$

Where:

$\overline{RT}$ Mean retention time, seconds
RT_i Retention time for the internal standard for each initial calibration standard, seconds.
n number of initial calibration levels

15.7.13 Equation Number 13

Percent Recovery (%R):

%R = X/TV x 100

where

X = Concentration of the analyte recovered
TV = True value of amount spiked

15.7.14 Equation Number 14

Relative Percent Difference (RPD)

$$RPD = \frac{|x_1 - x_2|}{\overline{x}}(100)$$

where:
x_1 First measurement value
x_2 Second measurement value
$\overline{x}$ Average of the two values

15.8 Data Review The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the data review checklist in Attachment 3. The data shall be reviewed and the sample results

calculated and assessed by one analyst and reviewed by a second qualified analyst. The data review checklist is used to document the reviews and once it has been completed, initialed and dated it must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.7.

15.9 Reporting The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and all information required by laboratory policy, TNI Standards, DoD QSM, and the TO-17 method including modifications, observances, data qualifiers, and certification information.

15.9.1 Analysis Observations / Case Narrative Summary Form This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved.

This form is necessary as a means of documentation. This form, among other information, will be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.9.2 Surrogate Only report surrogate results at the request of the client. If the surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 This section contains technical acceptance criteria and references to preferred corrective actions per the guidelines of the specified test method. To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.2 Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV. Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance Corrective Action*, where appropriate.

16.3 Instrument Performance Check

16.3.1 Acceptance Criteria

The GC/MS system must meet the mass spectra ion abundance criteria listed in Table 1. Results of the BFB tune check as well as any actual tuning must be recorded and a copy of the tune report maintained on file.

16.3.2 Corrective Action Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument's user manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria.

16.4 Initial Calibration

16.4.1 Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. Highest concentration, together with the lowest concentration, defines the calibration range.
3. Lowest concentration must be at or below the method reporting limit.
4. A blank should be analyzed prior to beginning the analysis of the calibration standards.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from low to high concentration.
8. All ICAL analyses, including the ICV, must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
    - Points at the high end may be dropped, but doing so lowers the calibration range.
    - Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).
    - Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.

      When an individual compound point is dropped from an ICAL both the response and concentration fields in the compound database of the method must be cleared. This ensures the average ICAL RRF calculates correctly when executing the CCV check routine.

- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
- Once the ICAL has been used to calculate and report sample results, it is not to be changed.

16.4.2 Acceptance Criteria Refer to the following acceptance criteria for the initial calibration.

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.
- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).
- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

16.4.3 Corrective Action Follow the initial calibration guidelines detailed in Section 16.4.1 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards. If the criteria are not met it may be necessary to perform maintenance, if this is the case then all calibration points must be re-analyzed.

16.5 Initial Calibration Verification Standard (ICV)

16.5.1 Acceptance Criteria The percent recovery for each compound in the ICV must be between 70%-130%.

16.5.2 Corrective Action If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it still fails prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. A demonstration of an acceptable ICV is required.

16.6 Continuing Calibration Verification (CCV)

16.6.1 Acceptance Criteria The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

16.6.2 Corrective Action If the continuing calibration verification technical acceptance criteria are not met, reanalyze once and if it still fails prepare a new spiking standard (if applicable) and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

> *When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects may be reported with the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative.*

16.6.3 DoD Requirement: If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (immediately is defined as within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.7 Method Blank

16.7.1 Acceptance Criteria

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the mid-level standard of the ICAL if the method blank follows the ICAL. If the method blank follows a CCV then the area response for each internal standard in the blank must be within ±40 percent of the area response of the CCV.
- The retention time for each internal standard in the blank must be within ±0.33 minutes of the retention time for each internal standard in the mid-level standard of the ICAL if the method blank follows the ICAL. If the method blank follows a CCV then the retention time for each internal standard in the blank must be within ±0.33 minutes of the retention time of the CCV.
- The method blank result for any target analyte cannot be greater than the reporting limit, AND be greater than 1/10th of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.

- For DoD samples, the method blank will be considered contaminated if:
  1. The concentration of any target analyte in the blank exceeds ½ the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
  2. The concentration of any common laboratory contaminant in the blank exceeds the LOQ;

16.7.2 Corrective Action If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with another pre-cleaned tube until results are acceptable or recondition the tube for about twenty minutes and reanalyze. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Determine whether the contamination is from the instrument or due to contamination in the blank tube (if results from the new or reconditioned tube are not acceptable then the system is probably contaminated). Regardless, appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.3.

If the requirements for the internal standard are not met then follow the corrective action detailed in Section 16.9.

16.8 Laboratory Control Sample (LCS)

16.8.1 Acceptance Criteria All analytes should have a 70-130% recovery and a RPD of 25% or less. Laboratory generated control limits must be generated when enough points are available.

16.8.2 Corrective Action If the LCS criteria are not met, determine whether the cause is instrumentation, the result of a poor injection, or a bad spiking stock standard. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. It may be necessary to prepare a new spiking stock. Also, the reporting requirements in Section 18.4 must be followed. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior; therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.9 Sample Analysis

16.9.1 Acceptance Criteria Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification. Samples out of holding time must be handled according to Section 16.11.

The following are the technical acceptance criteria for sample analysis:

- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in Sections 16.3 through 16.6.
- All target analyte peaks must be within the initial calibration range (Section 18.7.3) or reported with the appropriate data qualifier.
- The retention time for each internal standard must be within ±20 seconds of the retention time of the internal standard in the most recent valid calibration.

- Acceptable surrogate recoveries are based on a fixed window at 70 - 140%.
- The area response for any internal standard must be within the range of 60 - 140% of the area of the most recent valid calibration (CCV or mid-point from the initial calibration, whichever is most current).

Note 1: If the most recent valid calibration is an initial calibration, internal standard area responses and retention times in the sample are evaluated against the corresponding internal standard area responses and RTs in the mid-level standard of the initial calibration.

Note 2: Surrogate compound recoveries are requested by a number of clients and may be reported, but the use of surrogates is not addressed in Method TO-17 or TO-15. The surrogate compound is spiked onto the tubes at the same time as the internal standards, and is used as a system monitoring compound.

16.9.2 Corrective Action

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and note in the case narrative. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must have the appropriate data qualifiers attached to the results.

  If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for matrix interferences. If the response is high, it is likely that interference is present. If the response is low, it may be due to excessive co-collected water from the sample tube that was not sufficiently removed during the dry purge step. Report results with the appropriate qualifier. Reanalysis of the sample is usually not possible since the entire sample is consumed by the analysis.

Surrogate Results Poor surrogate recovery should be noted in the case narrative. Refer to Section 16.9.1 (Note 2) for specific information on the use of surrogates.

16.10 Sample's Holding Time Expired The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail

messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix H of the most recent version of the Quality Assurance Manual.

Note: No analyte results may be reported with an unacceptable initial calibration or initial calibration verification standard. However, any analyte not meeting such requirements (and the initial calibration is to be used) must be eliminated from the reporting list and any action taken fully documented.

18.2 Continuing Calibration Verification

- When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
- If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects may be reported with the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative. If this is the case then a full explanation must be noted in the case narrative of the final report. Refer to Section 15.9 for additional reporting requirements.

18.3 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.4 Laboratory Control Sample All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.5 Surrogate Refer to Section 16.9.1 (Note 2) for specific information on the use of these compounds. Report surrogate recoveries only at the request of the client and report sample results with the appropriate data qualifier.

18.6 Internal Standard All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.7 Estimated Sample Results

18.7.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

18.7.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.7.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported has having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

19.2 Method Detection Limit (MDL) The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV if needed.

19.3 Accuracy and Precision Refer to the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%.

19.4 Selectivity Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 33 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability An initial demonstration of capability shall be performed prior to analysis of samples and shall include the analysis of four (4) LCS spike standards at approximately four (4) times the MRL for each analyte with acceptable recoveries (as specified in this SOP). A continuing demonstration of proficiency must be performed annually per analyst and shall be conducted in the same manner as the initial demonstration.

19.6 Quarterly Demonstrations A demonstration of method sensitivity must be performed quarterly on each instrument performing sample analysis for DoD projects. A spike at the current LOD must be analyzed (Section 12.10) and verification of precision and bias at the LOQ demonstrated (Section 11.3).

19.7 Proficiency Testing (PT) Program The laboratory must participate in an air and emissions PT study for TO-17. The testing shall be performed in accordance with this document and the *SOP for Proficiency Testing Sample Analysis* and meet the frequency and proficiency requirements detailed in the DoD QSM.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
09.0	01/30/16	C. Humphrey	1.3 – Removed section. No longer applicable.
			12.4 – Revised bullets to add clarification to BFB tuning evaluation procedure
			12.8.2 – Added detailed procedure for reporting TICs
			14.1 – revised 2nd paragraph
			15.9 – Changed NELAC/TNI to TNI
			16.4.1 – Number 10 (5th bullet) added second paragraph
			Table 3 – updated concentrations

21) References and Related Documents

21.1 EPA Method TO-17, *Compendium of Methods for the Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes*, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.2 EPA Method TO-15, *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.3 *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition, January 1999.

21.4 *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition, Addendum, January 17, 2002.

21.5 *SOP for Batches and Sequences*, SOP ID ADM-BATCH_SEQ

21.6 *SOP for Making Entries onto Analytical Records*, SOP ID CE-QA007

21.7 *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*, SOP ID CE-QA011.

21.8 *SOP for Manual Integration Policy*, SOP ID CE-QA002

21.9 *SOP for Nonconformance and Corrective Action*, SOP ID CE-QA008

21.10 *SOP for Proficiency Testing Sample Analysis*, SOP ID CE-QA006

21.11 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.

21.12 TNI 2009 Standards

21.13 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

22) Appendix

22.1 Tables

Table 1 – Instrument Tune Check Ion Abundance Criteria (TO-15)

Table 2 – Volatile Organic Compounds, EPA Compendium Method TO-15
- Target Analytes
- CAS Numbers
- Primary Ions
- Secondary Ions
- Associated Internal Standards
- Molecular Weights

Table 3 – Standard Concentrations (Primary Source)

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Data Review Checklist

TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

TABLE 2

VOLATILE ORGANIC COMPOUNDS EPA COMPENDIUM METHOD TO-15					
Compound	CAS Number	Primary Ion[1]	Secondary Ion(s)[1]	MRL (ng/sample)[2]	Internal Standards/MW
Bromochloromethane (IS1)	74-97-5	130	128, 49	NA	-
Propene	115-07-1	42	39, 41	5.0	IS1/42.08
Dichlorodifluoromethane (CFC12)	75-71-8	85	87, 101, 103	0.5	IS1/120.9
Chloromethane	74-87-3	50	52	0.5	IS1 / 50.49
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	135	137	0.5	IS1 / 170.92
Vinyl Chloride	75-01-4	62	64	0.5	IS1 / 62.50
1,3-Butadiene	106-99-0	54	39, 53	1.0	IS1 / 54.09
Bromomethane	74-83-9	94	96	2.0	IS1 / 94.94
Chloroethane	75-00-3	64	66	1.0	IS1 / 64.52
Ethanol	64-17-5	45	46	5.0	IS1 / 46.07
Acetonitrile	75-05-8	41	40	2.0	IS1 / 41.05
Acrolein	107-02-8	56	55	2.0	IS1 / 56.06
Acetone	67-64-1	58	43	5.0	IS1 / 58.08
Trichlorofluoromethane	75-69-4	101	103	0.5	IS1 / 137.4
Isopropanol	67-63-0	45	43	2.0	IS1 / 60.10
Acrylonitrile	107-13-1	53	52	0.5	IS1 / 53.06
1,1-Dichloroethene	75-35-4	96	61	0.5	IS1 / 96.94
tert-Butanol	75-65-0	59	57, 41, 43	1.0	IS1 / 74.12
Methylene Chloride	75-09-2	84	49	0.5	IS1 / 84.94
Allyl Chloride	107-05-1	41	76	0.5	IS1 / 76.53
Trichlorotrifluoroethane	76-13-1	151	101	0.5	IS1 / 187.38
Carbon Disulfide	75-15-0	76	78	5.0	IS1 / 76.14
trans-1,2-Dichloroethene	156-60-5	61	96	0.5	IS1 / 96.94
1,1-Dichloroethane	75-34-3	63	65	0.5	IS1 / 98.96
Methyl tert-Butyl Ether	1634-04-4	73	57	0.5	IS1 / 88.15
2-Butanone	78-93-3	72	43	1.0	IS1 / 72.11
cis-1,2-Dichloroethene	156-59-2	61	96	0.5	IS1 / 96.94
n-Hexane	110-54-3	57	86	0.5	IS1 / 86.18

VOLATILE ORGANIC COMPOUNDS EPA COMPENDIUM METHOD TO-15					
Compound	CAS Number	Primary Ion[1]	Secondary Ion(s)[1]	MRL (ng/sample)[2]	Internal Standards/MW
Chloroform	67-66-3	83	85	0.5	IS1 / 119.4
1,2-Dichloroethane-d4(S)	17060-07-0	65	67	NA	IS1 / 102.98
Tetrahydrofuran	109-99-9	72	71,42	1.0	IS1 / 72.11
Ethyl tert-Butyl Ether	637-92-3	87	59, 57	0.50	IS1 / 102.176
1,2-Dichloroethane	107-06-2	62	64	0.50	IS1 / 98.96
1,4-Difluorobenzene(IS2)	540-36-3	114	63, 88	NA	-
1,1,1-Trichloroethane	71-55-6	97	99, 61	0.5	IS2 / 133.4
1-Butanol	71-36-3	56	41	2.0	IS2 / 74.1224
Benzene	71-43-2	78	77	2.0	IS2 / 78.11
Carbon Tetrachloride	56-23-5	117	119	0.5	IS2 / 153.8
Cyclohexane	110-82-7	84	69,56	1.0	IS2 / 84.16
tert-Amyl Methyl Ether	994-05-8	73	87, 55, 43	0.5	IS2 / 102.176
1,2-Dichloropropane	78-87-5	63	62	0.5	IS2 / 113
Bromodichloromethane	75-27-4	83	85	0.5	IS2 / 163.8
Trichloroethene	79-01-6	130	132	0.5	IS2 / 131.4
1,4-Dioxane	123-91-1	88	58	1.0	IS2 / 88.11
2,2,4-Trimethylpentane (Isooctane)	540-84-1	57	41	0.5	IS2 / 114.23
n-Heptane	142-82-5	71	57,100	0.5	IS2/100.2
cis-1,3-Dichloropropene	10061-01-5	75	77	0.5	IS2 / 111
4-Methyl-2-Pentanone	108-10-1	58	85	2.0	IS2 / 100.2
trans-1,3-Dichloropropene	10061-02-6	75	77	0.5	IS2 / 111
1,1,2-Trichloroethane	79-00-5	97	83	0.5	IS2 / 133.4
Chlorobenzene-d5(IS3)	3114-55-4	117	119, 82	NA	-
Toluene-d8(S)	2037-26-5	98	100	NA	IS3/100.19
Toluene	108-88-3	91	92	0.5	IS3 / 92.14
2-Hexanone	591-78-6	43	58	1.0	IS3 / 100.16
Dibromochloromethane	124-48-1	129	127	0.5	IS3 / 208.3
1,2-Dibromoethane	106-93-4	107	109	0.5	IS3 / 187.9
n-Octane	111-65-9	57	85, 71	0.5	IS3/114.23

VOLATILE ORGANIC COMPOUNDS EPA COMPENDIUM METHOD TO-15					
Compound	CAS Number	Primary Ion[1]	Secondary Ion(s)[1]	MRL (ng/sample)[2]	Internal Standards/MW
Tetrachloroethene	127-18-4	166	164	0.5	IS3 / 165.8
Chlorobenzene	108-90-7	112	114	0.5	IS3 / 112.6
Ethylbenzene	100-41-4	91	106	0.5	IS3 / 106.2
m-& p-Xylene	179601-23-1	91	106	1.0	IS3 / 106.2
Bromoform	75-25-2	173	175	0.5	IS3 / 252.8
Styrene	100-42-5	104	78, 103	0.5	IS3 / 104.1
o-Xylene	95-47-6	91	106	0.5	IS3 / 106.2
n-Nonane	111-84-2	43	57, 85, 128	0.5	IS3 / 128.26
1,1,2,2-Tetrachloroethane	79-34-5	83	85	0.5	IS3 / 167.9
4-Bromofluorobenzene(S)	460-00-4	174	176	NA	IS3/175
Cumene	98-82-8	105	120	0.5	IS3 / 120.2
Alpha-Pinene	80-56-8	93	77	0.5	IS3 / 136.24
n-Propylbenzene	103-65-1	91	120, 65	0.5	IS3 / 120.1938
3-Ethlytoluene	620-14-4	105	120	0.5	IS3 / 120.2
4-Ethyltoluene	622-96-8	105	120	0.5	IS3 / 120.2
1,3,5-Trimethylbenzene	108-67-8	105	120	0.5	IS3 / 120.2
Alpha-Methylstyrene	98-83-9	118	117, 103	0.5	IS3 / 118.19
2-Ethyltoluene	611-14-3	105	120	0.5	IS3 / 120.2
1,2,4-Trimethylbenzene	95-63-6	105	120	0.5	IS3 / 120.20
n-Decane	124-18-5	57	71, 85, 142	0.5	IS3 / 142.28
1,3-Dichlorobenzene	541-73-1	146	148	0.5	IS3 / 147
1,4-Dichlorobenzene	106-46-7	146	148	0.5	IS3 / 147
sec-Butylbenzene	135-98-8	105	134, 91	0.5	IS3 / 134.2206
p-Isopropyltoluene	99-87-6	119	134, 91	0.5	IS3 / 134.2206
1,2,3-Trimethylbenzene	526-73-8	105	120	0.5	IS3 / 120.1938
1,2-Dichlorobenzene	95-50-1	146	148	0.5	IS3 / 147
d-Limonene	5989-27-5	68	93	0.5	IS3 / 136.24
1,2,Dibromo-3-Chloropropane	96-12-8	157	75, 39	1.0	IS3 / 236.33
n-Undecane	1120-21-4	57	71, 85, 156	0.5	IS3 / 156.31
1,2,4-Trichlorobenzene	120-82-1	184	145, 182	0.5	IS3 / 181.5

VOLATILE ORGANIC COMPOUNDS EPA COMPENDIUM METHOD TO-15					
Compound	CAS Number	Primary Ion[1]	Secondary Ion(s)[1]	MRL (ng/sample)[2]	Internal Standards/MW
Naphthalene	91-20-3	128	129	0.5	IS3 / 128.17
n-Dodecane	112-40-3	57	71, 85, 170	0.5	IS3 / 170.34
Hexachloro-1,3-butadiene	87-68-3	225	227	0.5	IS3 / 260.8
Cyclohexanone	108-94-1	55	42, 98	5.0	IS3 / 98.14
tert-Butylbenzene	98-06-6	119	134	0.5	IS3 / 134.22
n-Butylbenzene	104-51-8	91	134	0.5	IS3 / 134.22

Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard limit (lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed.

Note 3: Additional compounds may be reported as long as the minimum requirements of this document are met.

Table 3 – Standard Concentrations (Primary Sources)

Compound Name	Density	0.5ng	1.0ng	2.0ng	5.0ng	10ng	20ng	50ng	100ng
	(g/mL)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)
Propene	-	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
Dichlorodifluoromethane	1.329	0.500	1.00	2.00	5.00	10.0	20.0	50.0	100
Chloromethane	0.911	0.490	0.98	1.96	4.90	9.8	19.6	49.0	98
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	1.455	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
Vinyl Chloride	0.9106	0.500	1.00	2.00	5.00	10.0	20.0	50.0	100
1,3-Butadiene	0.6149	0.530	1.06	2.12	5.30	10.6	21.2	53.0	106
Bromomethane	1.6755	0.500	1.00	2.00	5.00	10.0	20.0	50.0	100
Chloroethane	0.8902	0.505	1.01	2.02	5.05	10.1	20.2	50.5	101
Ethanol	0.7893	2.530	5.06	10.12	25.30	50.6	101.2	253.0	506
Acetonitrile	0.7857	0.510	1.02	2.04	5.10	10.2	20.4	51.0	102
Acrolein	0.840	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
Acetone	0.7845	2.685	5.37	10.74	26.85	53.7	107.4	268.5	537
Trichlorofluoromethane	-	0.495	0.99	1.98	4.95	9.9	19.8	49.5	99
Isopropanol	0.7809	1.045	2.09	4.18	10.45	20.9	41.8	104.5	209
Acrylonitrile	0.8060	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
1,1-Dichloroethene	1.213	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
tert-Butanol	0.7887	1.045	2.09	4.18	10.45	20.9	41.8	104.5	209
Methylene Chloride	1.3266	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
Allyl Chloride	0.9376	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
Trichlorotrifluoroethane	1.5635	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
Carbon Disulfide	1.2632	0.490	0.98	1.96	4.90	9.8	19.6	49.0	98
trans-1,2-Dichloroethene	1.2565	0.530	1.06	2.12	5.30	10.6	21.2	53.0	106
1,1-Dichloroethane	1.1757	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
Methyl tert-Butyl Ether	0.7402	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
Vinyl Acetate	0.9317	2.535	5.07	10.14	25.35	50.7	101.4	253.5	507
2-Butanone	0.7999	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
cis-1,2-Dichloroethene	1.2837	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
Diisopropyl Ether	0.7241	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
Ethyl Acetate	0.9003	1.060	2.12	4.24	10.60	21.2	42.4	106.0	212
n-Hexane	0.6548	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
Chloroform	1.4832	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
Tetrahydrofuran	0.8892	0.510	1.02	2.04	5.10	10.2	20.4	51.0	102
Ethyl tert-Butyl Ether	0.7519	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
1,2-Dichloroethane	1.2351	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
1,1,1-Trichloroethane	1.3390	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
Isopropyl acetate	0.8718	1.105	2.21	4.42	11.05	22.1	44.2	110.5	221
1-Butanol	0.8098	1.130	2.26	4.52	11.30	22.6	45.2	113.0	226
Benzene	0.8765	0.555	1.11	2.22	5.55	11.1	22.2	55.5	111
Carbon Tetrachloride	1.5940	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
Cyclohexane	0.7739	1.045	2.09	4.18	10.45	20.9	41.8	104.5	209
tert-Amyl Methyl Ether	0.7703	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104

Compound Name	Density	0.5ng	1.0ng	2.0ng	5.0ng	10ng	20ng	50ng	100ng
	(g/mL)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)
1,2-Dichloropropane	1.1560	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
Bromodichloromethane	1.980	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
Trichloroethene	1.4642	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
1,4-Dioxane	1.0337	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
2,2,4-Trimethylpentane (Isooctane)	0.6877	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
Methyl Methacrylate	0.944	1.040	2.08	4.16	10.40	20.8	41.6	104.0	208
n-Heptane	0.6837	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
cis-1,3-Dichloropropene	1.224	0.560	1.12	2.24	5.60	11.2	22.4	56.0	112
4-Methyl-2-Pentanone	0.7965	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
trans-1,3-Dichloropropene	1.217	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
1,1,2-Trichloroethane	1.4397	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
Toluene	0.8669	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
2-Hexanone	0.8113	0.555	1.11	2.22	5.55	11.1	22.2	55.5	111
Dibromochloromethane	2.451	0.550	1.10	2.20	5.50	11.0	22.0	55.0	110
1,2-Dibromoethane	2.1791	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
Butyl Acetate	0.8825	0.555	1.11	2.22	5.55	11.1	22.2	55.5	111
n-Octane	0.6986	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
Tetrachloroethene	1.6227	0.495	0.99	1.98	4.95	9.9	19.8	49.5	99
Chlorobenzene	1.1058	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
Ethylbenzene	0.8670	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
m- & p-Xylene	0.8642 0.8611	1.040	2.08	4.16	10.40	20.8	41.6	104.0	208
Bromoform	2.899	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
Styrene	0.9060	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
o-Xylene	0.8802	0.510	1.02	2.04	5.10	10.2	20.4	51.0	102
n-Nonane	0.7176	0.505	1.01	2.02	5.05	10.1	20.2	50.5	101
1,1,2,2-Tetrachloroethane	1.5953	0.500	1.00	2.00	5.00	10.0	20.0	50.0	100
Cumene	0.8618	0.505	1.01	2.02	5.05	10.1	20.2	50.5	101
alpha-Pinene	0.8582	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
n-Propylbenzene	0.8670	0.500	1.00	2.00	5.00	10.0	20.0	50.0	100
3-Ethyltoluene	0.8645	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
4-Ethyltoluene	0.8614	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
1,3,5-Trimethylbenzene	0.8652	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
Alpha-Methylstyrene	0.9106	0.515	1.03	2.06	5.15	10.3	20.6	51.5	103
2-Ethyltoluene	0.8807	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
1,2,4-Trimethylbenzene	0.8758	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
n-Decane	0.7300	0.505	1.01	2.02	5.05	10.1	20.2	50.5	101
Benzyl Chloride	1.1004	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
1,3-Dichlorobenzene	1.2884	0.540	1.08	2.16	5.40	10.8	21.6	54.0	108
1,4-Dichlorobenzene	1.2475	0.525	1.05	2.10	5.25	10.5	21.0	52.5	105
sec-Butylbenzene	0.8601	0.530	1.06	2.12	5.30	10.6	21.2	53.0	106
p-Isopropyltoluene	0.8573	0.500	1.00	2.00	5.00	10.0	20.0	50.0	100
1,2,3-Trimethylbenzene	0.8944	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
1,2-Dichlorobenzene	1.3059	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107

Compound Name	Density	0.5ng	1.0ng	2.0ng	5.0ng	10ng	20ng	50ng	100ng
	(g/mL)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)	Conc(ng)
d-Limonene	0.8402	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
1,2-Dibromo-3-Chloropropane	2.093	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
n-Undecane	0.7402	0.505	1.01	2.02	5.05	10.1	20.2	50.5	101
1,2,4-Trichlorobenzene	1.459	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
Naphthalene	1.0253	0.500	1.00	2.00	5.00	10.0	20.0	50.0	100
n-Dodecane	0.7487	0.520	1.04	2.08	5.20	10.4	20.8	52.0	104
Hexachloro-1,3-butadiene	1.556	0.535	1.07	2.14	5.35	10.7	21.4	53.5	107
Cyclohexanone	0.9478	0.560	1.12	2.24	5.60	11.2	22.4	56.0	112
tert-Butylbenzene	**0.867**	**0.525**	**1.05**	**2.10**	**5.25**	**10.5**	**21.0**	**52.5**	**105**
n-Butylbenzene	**0.867**	**0.540**	**1.08**	**2.16**	**5.40**	**10.8**	**21.6**	**54.0**	**108**

Note: The concentrations (for analytes in gas form) detailed in this table may change with each standard purchased. Refer to the appropriate initial calibration file for the corresponding concentrations.

Attachment 1

Training Plan

Training Plan for Analysis of VOCs by GC/MS

SOP Title: Determination of VOCs in Ambient Air using Sampling onto Sorbent Tubes

Trainee: _____ Trainer: _____ Instrument: _____

1. Read SOP — Trainer ____ Trainee ____ Date _____

2. Read Methods TO-15 and TO-17 — Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis — Trainer ____ Trainee ____ Date _____
   Whole air sample preconcentration techniques — *Training Duration* _____
   Gas chromatography
   Mass spectrometry

4. Demonstrated familiarity with related SOPs — Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences — *Training Duration* _____
   SOP for Making Entries onto Analytical Records
   SOP for Manual Integration Policy
   SOP for Significant Figures
   SOP for Nonconformance and Corrective Action
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation/initial calibration/initial calibration verification
   ___continuing calibration verification
   ___EnviroQuant introduction
   ___data reduction and reporting
   ___canister and tube handling

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation/initial calibration/initial calibration verification
   ___continuing calibration verification
   ___Sample analysis
   ___EnviroQuant use
   ___data reduction and reporting
   ___canister and tube handling

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample loading and sample dilutions
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation/initial calibration/initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant proficiency
   ___data reduction and reporting
   ___canister and tube handling
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance *Training Duration* _____Trainer ____ Trainee ____ Date _____
   ___autosampler ___mass spectrometer
   ___GC and capillary column installation ___data system

Attachment 2

Initial Calibration Checklist

Method: EPA TO-17 **ICAL Date:** _____ **Inst** ☐ MS5 ☐ MS18 ☐ MS20 **Mode** ☐ SIM ☐ Scan

ICAL ID: _____

Initial Calibration Review Checklist

Analyst		Reviewer
☐	1. Is the required documentation in the ICAL file?	☐
☐	BFB Tune analysis Report	☐
☐	Calibration Status Report (aka Calibration History)	☐
☐	Response Factor Report/Percent RSD	☐
☐	Print of the calibration curve for any compound that uses a curve (instead of average RF)	☐
☐	Quant Report for each calibration std (including manual integration documentation)	☐
☐	ICV Quantitation Report	☐
☐	Standard Calculation Spreadsheet	☐
☐	2. Was the ICAL performed continuously (i.e., not interrupted for maintenance or sample analysis)?	☐
☐	3. Have all the calibration standards been analyzed within 24 hours of each other?	☐
☐	4. Does the BFB tune check standard analysis at the start meet the tune criteria?	☐
☐	5. Are all the analytes in the blank analysis <MRL?	☐
☐	6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels?	☐
☐	7. Were the standards analyzed from low concentration to high concentration?	☐
☐	8. For each analyte, are there no levels skipped?	☐
☐	9. For each analyte, is there only one value used for each calibration level?	☐
☐	10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL?	☐
☐	11. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason?	☐
☐	12. Is the average RSD ≤30% for all analytes, with no more than two exceptions <40%?	☐
☐	13. Is the response Y at each calibration level within 40% of the mean area response over the Initial calibration range for each internal standard?	☐
☐	14. For the ICV analysis, is the percent recovery for each analyte 70%-130%?	☐
☐	15. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound?	☐
☐	16. Was the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard?	☐
☐	17. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages.	☐

COMMENTS:

Analyst: _____ Reviewer: _____

Attachment 3
Data Review Checklists

EPA Compendium Method TO-17 - Daily QC Review Checklist

(Note exceptions in Comments Section and attach Analysis Observations / Case Narrative Summary Form as appropriate)

Analysis Date: _____ Instrument: ☐MS5 ☐MS18 ☐MS20

Analysis Mode: ☐ SIM ☐ Scan DoD: ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation present?	☐
	☐ BFB Tune analysis Report	
	☐ CCV analysis Quantitation Report & Evaluate Continuing Calibration Report (Percent Diff. Report)	
	☐ LCS/LCSD analysis Quantitation Report	
	☐ MB analysis Quantitation Report	
☐	2. Does the BFB **tune** check standard analysis meet the tune criteria?	☐
☐	3. Are all analyses within the tune's **24-hr. window**?	☐
☐	4. Does the **CCV** have a difference ≤30% for all analytes? [Note outliers biased high and/or low]	☐
☐	5. Are all the **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)?	☐
☐	6. Are all the **IS** responses within ±40% of CCV or the midpoint in the ICAL?	☐
☐	7. Are all the **surrogate** recoveries (in CCVs, MBs, LCSs, etc.) within acceptance limits (70%-140%)?	☐
☐	8. Are all the analytes in the **MB** <MRL? (DoD <1/2 MRL, except common lab contaminants?)	☐
☐	9. Are all the analytes in the **LCS and LCSD** within 70%-130% recovery?	☐
☐	10. Is the RPD between the **LCS and LCSD** within 25%?	☐
☐	11. Are all peak **integrations** acceptable?	☐
☐	12. Are all manual **integrations** flagged and documented?	☐
☐	13. Has the analyst initialed and dated each **quantitation report**?	☐

COMMENTS:

Analyst/LIMS Run Approval: _____ Secondary/LIMS Supervisor Approval: _____

EPA Compendium Method TO-17 - Sample Review Checklist
(Note exceptions in Comments Section and attach Analysis Observations / Case Narrative Summary Form as appropriate)

Analysis Date: _____ Project #: _____

Instrument: ☐MS5 ☐MS18 ☐MS20 Analysis Mode: ☐ SIM ☐ Scan DoD: ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Are **quantitation reports** included for each sample?	☐
☐	2. Has the analyst initialed and dated each **quantitation report**?	☐
☐	3. Are all the analyte hits in the samples within the **calibration range** and/or noted?	☐
☐	4. Are all peak **integrations** acceptable?	☐
☐	5. Are all manual **integrations** flagged and documented?	☐
☐	6. Are spectral match details for each hit included, where necessary?	☐
☐	7. Are all **calculations** correct?	☐
☐	8. For **TICs** are the relative intensity and other requirements met?	☐
☐	9. Are the correct volumes entered on the **TIC report**?	☐
☐	10. **DoD:** Are **manual integrations** notated in the **case narrative**?	☐

COMMENTS:

Analyst/LIMS Run Approval: _____ Secondary/LIMS Supervisor Approval: _____