ATTACHMENT A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625

19. REQUIREMENTS FOR METHOD 625

19.1. Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table A-1.

19.2. This method can be applied only to aqueous matrices.

19.3. EPA has approved modification to method 625: one extraction can be done. In using single pH extractions for 625 the laboratory should analyze a series of LCSs and have the recovery and precision data filed and readily available. Refer to Appendix A.

19.4. The tune period for this method is defined as 24 hours.

19.5. Initial calibration curve requirements:

19.5.1. The initial calibration curve for this method requires at least three points.

19.5.2. Target compounds must have RSD ≤ 35%.

19.5.3. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.

19.6. Continuing calibration verification requirements: All target compounds must have %D ≤ 20%.

19.7. Matrix Spike and LCS requirements:

19.7.1. A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

19.8. The laboratory must, on an ongoing basis, spike at least 5% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to 20 samples per month, at least one spiked sample per month is required. The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control.

19.8.1. If any parameter fails the acceptance criteria for recovery, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of single-component parameters in must be measured in the sample, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spike sample.

Table A-1

TestAmerica Pittsburgh Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50

Table A-1

TestAmerica Pittsburgh Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous µg/L
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2
Method 625 LCS and MS Compounds and Spike Concentrations

LCS Compounds	Spiking Level, ng in injected 2 µL injection
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100

Table A-2
Method 625 LCS and MS Compounds and Spike Concentrations

LCS Compounds	Spiking Level, ng in injected 2 µL injection
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

TABLE A-3
Method 625 LCS and MS Compounds and Spike Concentrations

Surrogate Compounds	Spiking Level, ug/L in extract
Nitrobenzene-d5	50
2-Fluorobiphenyl	50
Terphenyl-d14	50
2-Fluorophenol	50
2,4,6-Tribromophenol	50
Phenol-d_5	50

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

TABLE A-4. METHOD 625 LCS, MS AND SURROGATE QC ACCEPTANCE CRITERIA

		LCS			Matrix Spike		
Compound	**MDL[1] (ug/L)**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
1,2,4-Trichlorobenzene	1.377	31	110	37	31	110	37
1,2-Dichlorobenzene	1.361	32	129	20	32	129	20
1,2-Diphenylhydrazine	1.339	30	125	25	30	125	25
1,3-Dichlorobenzene	1.269	1	172	35	1	172	35
1,4-Dichlorobenzene	1.317	28	110	36	28	110	36
2,2'-oxybis(1-Chloropropane)	1.715	50	150	50	50	150	50
2,4,6-Trichlorophenol	1.497	46	135	27	46	135	27
2,4-Dichlorophenol	1.335	42	115	44	42	115	44
2,4-Dimethylphenol	1.833	32	119	20	32	119	20
2,4-Dinitrophenol	14.763	1	191	53	1	191	53
2,4-Dinitrotoluene	1.289	47	131	32	47	131	32
2,6-Dinitrotoluene	1.4	50	158	20	50	158	20
2-Chloronaphthalene	1.429	60	118	20	60	118	20
2-Chlorophenol	1.389	19	124	43	19	124	43
2-Methyl-4,6-dinitrophenol	9.644	10	181	40	10	181	40
2-Nitrophenol	2.99	29	182	32	29	182	32
3,3'-Dichlorobenzidine	25.023	1	162	56	1	162	56
4-Bromophenyl phenyl ether	1.238	53	127	20	53	127	20
4-Chloro-3-methylphenol	1.314	29	124	55	29	124	55
4-Chlorophenyl phenyl ether	1.629	25	158	27	25	158	27
4-Nitrophenol	1.775	19	144	34	19	144	34
Acenaphthene	1.556	39	118	35	39	118	35
Acenaphthylene	1.822	33	145	23	33	145	23
Anthracene	1.195	27	133	22	27	133	22
Benzidine	1.998	1	140	50	1	140	50
Benzo(a)anthracene	0.932	33	143	23	33	143	23

TABLE A-4. METHOD 625 LCS, MS AND SURROGATE QC ACCEPTANCE CRITERIA

		LCS			Matrix Spike		
Compound	**MDL[1] (ug/L)**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Benzo(a)pyrene	2.503	17	163	31	17	163	31
Benzo(b)fluoranthene	0.857	24	159	28	24	159	28
Benzo(ghi)perylene	0.997	1	219	50	1	219	50
Benzo(k)fluoranthene	1.101	11	162	31	11	162	31
bis(2-Chloroethoxy)methane	3.45	33	184	30	33	184	30
bis(2-Chloroethyl) ether	1.44	12	158	30	12	158	30
bis(2-Ethylhexyl) phthalate	0.907	8	158	31	8	158	31
Butyl benzyl phthalate	1.011	1	152	35	1	152	35
Chrysene	0.953	17	168	31	17	168	31
Dibenzo(a,h)anthracene	1.039	1	227	55	1	227	55
Diethyl phthalate	1.13	1	114	24	1	114	24
Dimethyl phthalate	1.251	1	112	22	1	112	22
Di-n-butyl phthalate	1.104	1	118	24	1	118	24
Di-n-octyl phthalate	0.948	4	146	29	4	146	29
Fluoranthene	1.124	26	137	23	26	137	23
Fluorene	1.548	59	121	20	59	121	20
Hexachlorobenzene	1.261	57	128	22	57	128	22
Hexachlorobutadiene	1.47	36	116	32	36	116	32
Hexachlorocyclopentadiene	6.259	1	138	54	1	138	54
Hexachloroethane	1.371	30	110	33	30	110	33
Indeno(1,2,3-cd)pyrene	0.998	1	171	37	1	171	37
Isophorone	1.404	21	196	37	21	196	37
Naphthalene	1.5	21	133	23	21	133	23
Nitrobenzene	1.455	45	130	50	45	130	50
N-Nitrosodimethylamine	1.694	1	230	47	1	230	47
N-Nitrosodi-n-propylamine	1.538	30	115	36	30	115	36

TABLE A-4. METHOD 625 LCS, MS AND SURROGATE QC ACCEPTANCE CRITERIA

		LCS			Matrix Spike		
Compound	**MDL[1] (ug/L)**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
N-Nitrosodiphenylamine	4.191	5	138	68	5	138	68
Pentachlorophenol	0.816	10	140	56	10	140	56
Phenanthrene	1.068	54	120	20	54	120	20
Phenol	1.98	10	131	43	10	131	43
Pyrene	0.941	46	130	31	46	130	31
Surrogates:							
2-Fluorobiphenyl		30	110		30	110	
2-Fluorophenol		13	110		13	110	
2,4,6-Tribromophenol		21	122		21	122	
Nitrobenzene-d5		32	112		32	112	
Phenol-d5		10	113		10	113	
Terphenyl-d14		10	144		10	144	

Note: The control limits are derived from laboratory generated data.

[1] The MDL listed are subject to change.

Attachment B
Standard Preparation

TestAmerica Pittsburgh

Standards Preparation Logbook Record

Sep-09-2009

Logbook: \\pittvr01\stdslog\GCMS Semi-Volatile.std

BNA0890-09, +LLSSTD0 4(0.2ug/ml) Analyst: piccolinov

Solvent: Methylene chloride Lot No.: BNA0803-09 Volume (ml): 1.0000
Date Prep./Opened: 07-01-2009
Date Expires(1): 08-31-2009 (6 Months)
Date Reviewed: 08-18-2009 by bozike

Parent Std No.: BNA0854-08, 4,6dn2mphenol 100ug/ml Aliquot Amount (ml): 0.0080

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4,6-Dinitro-2-methylphenol	100.00	0.8000

Parent Std No.: BNA0855-08, 4 nitrophenol 100ug/ml Aliquot Amount (ml): 0.0080

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4-Nitrophenol	20.000	0.1600

Parent Std No.: BNA0856-08, 2,4 dinitrophenol 100ug/ml Aliquot Amount (ml): 0.0080

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
2,4-Dinitrophenol	20.000	0.1600

Parent Std No.: BNA0857-08, benzoic acid 100ug/ml Aliquot Amount (ml): 0.0080

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Benzoic Acid	100.00	0.8000

Parent Std No.: BNA0858-08, pentachlorophenol 100ug/ml Aliquot Amount (ml): 0.0080

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Pentachlorophenol	100.00	0.8000

Parent Std No.: BNA0866-09, LOW LEVEL Stock 40ug/ml Aliquot Amount (ml): 0.0050

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Indene	40.000	0.2000
2,4,6-Tribromophenol	40.000	0.2000
2-Chlorophenol-d4	40.000	0.2000
2-Fluorophenol	40.000	0.2000
Phenol-d5	40.000	0.2000
1,2-Dichlorobenzene-d4	40.000	0.2000
2-Fluorobiphenyl	40.000	0.2000
Nitrobenzene-d5	40.000	0.2000
p-Terphenyl-d14	40.000	0.2000

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3,3'-Dichlorobenzidine	40.000	0.2000
3,3'-dimethylbenzidine	40.000	0.2000
Benzidine	40.000	0.2000
1-Methylnaphthalene	40.000	0.2000
2,3,4,6-Tetrachlorophenol	40.000	0.2000
2,3,5,6-Tetrachlorophenol	40.000	0.2000
2-Naphthylamine	40.000	0.2000
7,12-Dimethylbenz(a)anthracene	40.000	0.2000
Methyl methanesulfonate	40.000	0.2000
N-Nitroso-di-n-butylamine	40.000	0.2000
1,4 Dioxane	40.000	0.2000
1,2,4,5-Tetrachlorobenzene	40.000	0.2000
Acetophenone	40.000	0.2000
Atrazine	40.000	0.2000
Benzaldehyde	40.000	0.2000
Biphenyl	40.000	0.2000
Caprolactam	40.000	0.2000
1-Nitrosopyrrolidine	40.000	0.2000
Aniline	40.000	0.2000
Benzoic Acid	40.000	0.2000
Benzyl Alcohol	40.000	0.2000
Pyridine	40.000	0.2000
N-Nitrosodiphenylamine	40.000	0.2000
2,6-Dichlorophenol	40.000	0.2000
1,2,4-Trichlorobenzene	40.000	0.2000
1,2-Dichlorobenzene	40.000	0.2000
1,3-Dichlorobenzene	40.000	0.2000
1,4-Dichlorobenzene	40.000	0.2000
2,4,5-Trichlorophenol	40.000	0.2000
2,4,6-Trichlorophenol	40.000	0.2000
2,4-Dichlorophenol	40.000	0.2000
2,4-Dimethylphenol	40.000	0.2000
2,4-Dinitrophenol	40.000	0.2000
2,4-Dinitrotoluene	40.000	0.2000
2,6-Dinitrotoluene	40.000	0.2000
2-Chloroisopropyl Ether	40.000	0.2000
2-Chloronaphthalene	40.000	0.2000
2-Chlorophenol	40.000	0.2000
2-Methyl-4,6-Dinitrophenol	40.000	0.2000
2-Methylnaphthalene	40.000	0.2000
2-Nitroaniline	40.000	0.2000
2-Nitrophenol	40.000	0.2000
3-Nitroaniline	40.000	0.2000
4-Bromodiphenyl Ether	40.000	0.2000
4-Chloro-3-Methylphenol	40.000	0.2000
4-Chloroaniline	40.000	0.2000
4-Chlorodiphenyl Ether	40.000	0.2000
4-Nitroaniline	40.000	0.2000
4-Nitrophenol	40.000	0.2000
Acenaphthene	40.000	0.2000
Acenaphthylene	40.000	0.2000

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Anthracene	40.000	0.2000
Azobenzene	40.000	0.2000
Benz(A)Anthracene	40.000	0.2000
Benzo(A)Pyrene	40.000	0.2000
Benzo(B)Fluoranthene	40.000	0.2000
Benzo(ghi)Perylene	40.000	0.2000
Benzo(K)Fluoranthene	40.000	0.2000
Benzyl Butyl Phthalate	40.000	0.2000
Bis(2-Chloroethoxy)Methane	40.000	0.2000
Bis(2-Chloroethyl)Ether	40.000	0.2000
Bis(2-Ethylhexyl)Phthalate	40.000	0.2000
Carbazole	40.000	0.2000
Chrysene	40.000	0.2000
Dibenz(A,H)Anthracene	40.000	0.2000
Dibenzofuran	40.000	0.2000
Dibutyl Phthalate	40.000	0.2000
Diethyl Phthalate	40.000	0.2000
Dimethyl Phthalate	40.000	0.2000
Dioctyl Phthalate	40.000	0.2000
Fluoranthene	40.000	0.2000
Fluorene	40.000	0.2000
Hexachloro-1,3-butadiene	40.000	0.2000
Hexachlorobenzene	40.000	0.2000
Hexachlorocyclopentadiene	40.000	0.2000
Hexachloroethane	40.000	0.2000
Indeno(1,2,3-cd)Pyrene	40.000	0.2000
Isophorone	40.000	0.2000
N-Nitrosodi-N-propylamine	40.000	0.2000
N-Nitrosodimethylamine	40.000	0.2000
Naphthalene	40.000	0.2000
Nitrobenzene	40.000	0.2000
O-Cresol	40.000	0.2000
P-Cresol	40.000	0.2000
Pentachlorophenol	40.000	0.2000
Phenanthrene	40.000	0.2000
Phenol	40.000	0.2000
Pyrene	40.000	0.2000

Reviewed By: _____

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TestAmerica Pittsburgh

Standards Preparation Logbook Record

Sep-09-2009

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0891-09, +LLSSTD2.0(1.0ug/ml) Analyst: piccolinov

Solvent: Methylene chloride Lot No.: BNA0803-09 Volume (ml): 1.0000
Date Prep./Opened: 07-01-2009
Date Expires(1): 08-31-2009 (6 Months)
Date Reviewed: 08-18-2009 by bozike

Parent Std No.: BNA0854-08, 4,6dn2mphenol 100ug/ml Aliquot Amount (ml): 0.0100

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4,6-Dinitro-2-methylphenol	100.00	1.0000

Parent Std No.: BNA0855-08, 4 nitrophenol 100ug/ml Aliquot Amount (ml): 0.0100

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4-Nitrophenol	20.000	0.2000

Parent Std No.: BNA0856-08, 2,4 dinitrophenol 100ug/ml Aliquot Amount (ml): 0.0100

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
2,4-Dinitrophenol	20.000	0.2000

Parent Std No.: BNA0857-08, benzoic acid 100ug/ml Aliquot Amount (ml): 0.0100

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Benzoic Acid	100.00	1.0000

Parent Std No.: BNA0858-08, pentachlorophenol 100ug/ml Aliquot Amount (ml): 0.0100

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Pentachlorophenol	100.00	1.0000

Parent Std No.: BNA0866-09, LOW LEVEL Stock 40ug/ml Aliquot Amount (ml): 0.0250

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Benzyl Alcohol	40.000	1.0000
Pyridine	40.000	1.0000
N-Nitrosodiphenylamine	40.000	1.0000
2,6-Dichlorophenol	40.000	1.0000
1,2,4-Trichlorobenzene	40.000	1.0000
1,2-Dichlorobenzene	40.000	1.0000
1,3-Dichlorobenzene	40.000	1.0000
1,4-Dichlorobenzene	40.000	1.0000
2,4,5-Trichlorophenol	40.000	1.0000

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2,4,6-Trichlorophenol	40.000	1.0000
2,4-Dichlorophenol	40.000	1.0000
2,4-Dimethylphenol	40.000	1.0000
2,4-Dinitrophenol	40.000	1.0000
2,4-Dinitrotoluene	40.000	1.0000
2,6-Dinitrotoluene	40.000	1.0000
2-Chloroisopropyl Ether	40.000	1.0000
2-Chloronaphthalene	40.000	1.0000
2-Chlorophenol	40.000	1.0000
2-Methyl-4,6-Dinitrophenol	40.000	1.0000
2-Methylnaphthalene	40.000	1.0000
2-Nitroaniline	40.000	1.0000
2-Nitrophenol	40.000	1.0000
3-Nitroaniline	40.000	1.0000
4-Bromodiphenyl Ether	40.000	1.0000
4-Chloro-3-Methylphenol	40.000	1.0000
4-Chloroaniline	40.000	1.0000
4-Chlorodiphenyl Ether	40.000	1.0000
4-Nitroaniline	40.000	1.0000
4-Nitrophenol	40.000	1.0000
Acenaphthene	40.000	1.0000
Acenaphthylene	40.000	1.0000
Anthracene	40.000	1.0000
Azobenzene	40.000	1.0000
Benz(A)Anthracene	40.000	1.0000
Benzo(A)Pyrene	40.000	1.0000
Benzo(B)Fluoranthene	40.000	1.0000
Benzo(ghi)Perylene	40.000	1.0000
Benzo(K)Fluoranthene	40.000	1.0000
Benzyl Butyl Phthalate	40.000	1.0000
Bis(2-Chloroethoxy)Methane	40.000	1.0000
Bis(2-Chloroethyl)Ether	40.000	1.0000
Bis(2-Ethylhexyl)Phthalate	40.000	1.0000
Carbazole	40.000	1.0000
Chrysene	40.000	1.0000
Dibenz(A,H)Anthracene	40.000	1.0000
Dibenzofuran	40.000	1.0000
Dibutyl Phthalate	40.000	1.0000
Diethyl Phthalate	40.000	1.0000
Dimethyl Phthalate	40.000	1.0000
Dioctyl Phthalate	40.000	1.0000
Fluoranthene	40.000	1.0000
Fluorene	40.000	1.0000
Hexachloro-1,3-butadiene	40.000	1.0000
Hexachlorobenzene	40.000	1.0000
Hexachlorocyclopentadiene	40.000	1.0000
Hexachloroethane	40.000	1.0000
Indeno(1,2,3-cd)Pyrene	40.000	1.0000
Isophorone	40.000	1.0000
N-Nitrosodi-N-propylamine	40.000	1.0000
N-Nitrosodimethylamine	40.000	1.0000

Reviewed By: _____

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Naphthalene	40.000	1.0000
Nitrobenzene	40.000	1.0000
O-Cresol	40.000	1.0000
P-Cresol	40.000	1.0000
Pentachlorophenol	40.000	1.0000
Indene	40.000	1.0000
2,4,6-Tribromophenol	40.000	1.0000
2-Chlorophenol-d4	40.000	1.0000
2-Fluorophenol	40.000	1.0000
Phenol-d5	40.000	1.0000
1,2-Dichlorobenzene-d4	40.000	1.0000
2-Fluorobiphenyl	40.000	1.0000
Nitrobenzene-d5	40.000	1.0000
p-Terphenyl-d14	40.000	1.0000
3,3'-Dichlorobenzidine	40.000	1.0000
3,3'-dimethylbenzidine	40.000	1.0000
Benzidine	40.000	1.0000
1-Methylnaphthalene	40.000	1.0000
2,3,4,6-Tetrachlorophenol	40.000	1.0000
2,3,5,6-Tetrachlorophenol	40.000	1.0000
2-Naphthylamine	40.000	1.0000
7,12-Dimethylbenz(a)anthracene	40.000	1.0000
Methyl methanesulfonate	40.000	1.0000
N-Nitroso-di-n-butylamine	40.000	1.0000
1,4 Dioxane	40.000	1.0000
1,2,4,5-Tetrachlorobenzene	40.000	1.0000
Acetophenone	40.000	1.0000
Atrazine	40.000	1.0000
Benzaldehyde	40.000	1.0000
Biphenyl	40.000	1.0000
Caprolactam	40.000	1.0000
!-Nitrosopyrrolidine	40.000	1.0000
Aniline	40.000	1.0000
Benzoic Acid	40.000	1.0000
Phenanthrene	40.000	1.0000
Phenol	40.000	1.0000
Pyrene	40.000	1.0000

Reviewed By: _____

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TestAmerica Pittsburgh

Standards Preparation Logbook Record

Sep-09-2009

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0974-09, +LLSSTD4.0(2.0ug/ml) Analyst: bungardf

Solvent: Methylene chloride
Date Prep./Opened: 08-09-2009
Date Expires(1): 08-16-2009 (1 Week)
Volume (ml): 1.0000

Parent Std No.: BNA0854-08, 4,6dn2mphenol 100ug/ml Aliquot Amount (ml): 0.0300

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4,6-Dinitro-2-methylphenol	100.00	3.0000

Parent Std No.: BNA0855-08, 4 nitrophenol 100ug/ml Aliquot Amount (ml): 0.0300

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4-Nitrophenol	20.000	0.6000

Parent Std No.: BNA0856-08, 2,4 dinitrophenol 100ug/ml Aliquot Amount (ml): 0.0300

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
2,4-Dinitrophenol	20.000	0.6000

Parent Std No.: BNA0857-08, benzoic acid 100ug/ml Aliquot Amount (ml): 0.0300

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Benzoic Acid	100.00	3.0000

Parent Std No.: BNA0858-08, pentachlorophenol 100ug/ml Aliquot Amount (ml): 0.0300

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Pentachlorophenol	100.00	3.0000

Parent Std No.: BNA0866-09, LOW LEVEL Stock 40ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Indene	40.000	2.0000
2,4,6-Tribromophenol	40.000	2.0000
2-Chlorophenol-d4	40.000	2.0000
2-Fluorophenol	40.000	2.0000
Phenol-d5	40.000	2.0000
1,2-Dichlorobenzene-d4	40.000	2.0000
2-Fluorobiphenyl	40.000	2.0000
Nitrobenzene-d5	40.000	2.0000
p-Terphenyl-d14	40.000	2.0000
3,3'-Dichlorobenzidine	40.000	2.0000

Reviewed By: _____

Page 1 of 3

3,3'-dimethylbenzidine	40.000	2.0000
Benzidine	40.000	2.0000
1-Methylnaphthalene	40.000	2.0000
2,3,4,6-Tetrachlorophenol	40.000	2.0000
2,3,5,6-Tetrachlorophenol	40.000	2.0000
2-Naphthylamine	40.000	2.0000
7,12-Dimethylbenz(a)anthracene	40.000	2.0000
Methyl methanesulfonate	40.000	2.0000
N-Nitroso-di-n-butylamine	40.000	2.0000
1,4 Dioxane	40.000	2.0000
1,2,4,5-Tetrachlorobenzene	40.000	2.0000
Acetophenone	40.000	2.0000
Atrazine	40.000	2.0000
Benzaldehyde	40.000	2.0000
Biphenyl	40.000	2.0000
Caprolactam	40.000	2.0000
1-Nitrosopyrrolidine	40.000	2.0000
Aniline	40.000	2.0000
Benzoic Acid	40.000	2.0000
Benzyl Alcohol	40.000	2.0000
Pyridine	40.000	2.0000
N-Nitrosodiphenylamine	40.000	2.0000
2,6-Dichlorophenol	40.000	2.0000
1,2,4-Trichlorobenzene	40.000	2.0000
1,2-Dichlorobenzene	40.000	2.0000
1,3-Dichlorobenzene	40.000	2.0000
1,4-Dichlorobenzene	40.000	2.0000
2,4,5-Trichlorophenol	40.000	2.0000
2,4,6-Trichlorophenol	40.000	2.0000
2,4-Dichlorophenol	40.000	2.0000
2,4-Dimethylphenol	40.000	2.0000
2,4-Dinitrophenol	40.000	2.0000
2,4-Dinitrotoluene	40.000	2.0000
2,6-Dinitrotoluene	40.000	2.0000
2-Chloroisopropyl Ether	40.000	2.0000
2-Chloronaphthalene	40.000	2.0000
2-Chlorophenol	40.000	2.0000
2-Methyl-4,6-Dinitrophenol	40.000	2.0000
2-Methylnaphthalene	40.000	2.0000
2-Nitroaniline	40.000	2.0000
2-Nitrophenol	40.000	2.0000
3-Nitroaniline	40.000	2.0000
4-Bromodiphenyl Ether	40.000	2.0000
4-Chloro-3-Methylphenol	40.000	2.0000
4-Chloroaniline	40.000	2.0000
4-Chlorodiphenyl Ether	40.000	2.0000
4-Nitroaniline	40.000	2.0000
4-Nitrophenol	40.000	2.0000
Acenaphthene	40.000	2.0000
Acenaphthylene	40.000	2.0000
Anthracene	40.000	2.0000

Reviewed By: _____

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Azobenzene	40.000	2.0000
Benz(A)Anthracene	40.000	2.0000
Benzo(A)Pyrene	40.000	2.0000
Benzo(B)Fluoranthene	40.000	2.0000
Benzo(ghi)Perylene	40.000	2.0000
Benzo(K)Fluoranthene	40.000	2.0000
Benzyl Butyl Phthalate	40.000	2.0000
Bis(2-Chloroethoxy)Methane	40.000	2.0000
Bis(2-Chloroethyl)Ether	40.000	2.0000
Bis(2-Ethylhexyl)Phthalate	40.000	2.0000
Carbazole	40.000	2.0000
Chrysene	40.000	2.0000
Dibenz(A,H)Anthracene	40.000	2.0000
Dibenzofuran	40.000	2.0000
Dibutyl Phthalate	40.000	2.0000
Diethyl Phthalate	40.000	2.0000
Dimethyl Phthalate	40.000	2.0000
Dioctyl Phthalate	40.000	2.0000
Fluoranthene	40.000	2.0000
Fluorene	40.000	2.0000
Hexachloro-1,3-butadiene	40.000	2.0000
Hexachlorobenzene	40.000	2.0000
Hexachlorocyclopentadiene	40.000	2.0000
Hexachloroethane	40.000	2.0000
Indeno(1,2,3-cd)Pyrene	40.000	2.0000
Isophorone	40.000	2.0000
N-Nitrosodi-N-propylamine	40.000	2.0000
N-Nitrosodimethylamine	40.000	2.0000
Naphthalene	40.000	2.0000
Nitrobenzene	40.000	2.0000
O-Cresol	40.000	2.0000
P-Cresol	40.000	2.0000
Pentachlorophenol	40.000	2.0000
Phenanthrene	40.000	2.0000
Phenol	40.000	2.0000
Pyrene	40.000	2.0000

Reviewed By: _____

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TestAmerica Pittsburgh

Standards Preparation Logbook Record

Sep-09-2009

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0892-09, +LLSSTD10(5ug/ml) Analyst: piccolinov

Solvent: Methylene chloride Lot No.: BNA0803-09 Volume (ml): 1.0000
Date Prep./Opened: 07-01-2009
Date Expires(1): 08-31-2009 (6 Months)
Date Reviewed: 08-18-2009 by bozike

Parent Std No.: BNA0854-08, 4,6dn2mphenol 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4,6-Dinitro-2-methylphenol	100.00	5.0000

Parent Std No.: BNA0855-08, 4 nitrophenol 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4-Nitrophenol	20.000	1.0000

Parent Std No.: BNA0856-08, 2,4 dinitrophenol 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
2,4-Dinitrophenol	20.000	1.0000

Parent Std No.: BNA0857-08, benzoic acid 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Benzoic Acid	100.00	5.0000

Parent Std No.: BNA0858-08, pentachlorophenol 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Pentachlorophenol	100.00	5.0000

Parent Std No.: BNA0866-09, LOW LEVEL Stock 40ug/ml Aliquot Amount (ml): 0.1250

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Indene	40.000	5.0000
2,4,6-Tribromophenol	40.000	5.0000
2-Chlorophenol-d4	40.000	5.0000
2-Fluorophenol	40.000	5.0000
Phenol-d5	40.000	5.0000
1,2-Dichlorobenzene-d4	40.000	5.0000
2-Fluorobiphenyl	40.000	5.0000
Nitrobenzene-d5	40.000	5.0000
p-Terphenyl-d14	40.000	5.0000

Reviewed By: _____

Page 1 of 3

3,3'-Dichlorobenzidine	40.000	5.0000
3,3'-dimethylbenzidine	40.000	5.0000
Benzidine	40.000	5.0000
1-Methylnaphthalene	40.000	5.0000
2,3,4,6-Tetrachlorophenol	40.000	5.0000
2,3,5,6-Tetrachlorophenol	40.000	5.0000
2-Naphthylamine	40.000	5.0000
7,12-Dimethylbenz(a)anthracene	40.000	5.0000
Methyl methanesulfonate	40.000	5.0000
N-Nitroso-di-n-butylamine	40.000	5.0000
1,4 Dioxane	40.000	5.0000
1,2,4,5-Tetrachlorobenzene	40.000	5.0000
Acetophenone	40.000	5.0000
Atrazine	40.000	5.0000
Benzaldehyde	40.000	5.0000
Biphenyl	40.000	5.0000
Caprolactam	40.000	5.0000
1-Nitrosopyrrolidine	40.000	5.0000
Aniline	40.000	5.0000
Benzoic Acid	40.000	5.0000
Benzyl Alcohol	40.000	5.0000
Pyridine	40.000	5.0000
N-Nitrosodiphenylamine	40.000	5.0000
2,6-Dichlorophenol	40.000	5.0000
1,2,4-Trichlorobenzene	40.000	5.0000
1,2-Dichlorobenzene	40.000	5.0000
1,3-Dichlorobenzene	40.000	5.0000
1,4-Dichlorobenzene	40.000	5.0000
2,4,5-Trichlorophenol	40.000	5.0000
2,4,6-Trichlorophenol	40.000	5.0000
2,4-Dichlorophenol	40.000	5.0000
2,4-Dimethylphenol	40.000	5.0000
2,4-Dinitrophenol	40.000	5.0000
2,4-Dinitrotoluene	40.000	5.0000
2,6-Dinitrotoluene	40.000	5.0000
2-Chloroisopropyl Ether	40.000	5.0000
2-Chloronaphthalene	40.000	5.0000
2-Chlorophenol	40.000	5.0000
2-Methyl-4,6-Dinitrophenol	40.000	5.0000
2-Methylnaphthalene	40.000	5.0000
2-Nitroaniline	40.000	5.0000
2-Nitrophenol	40.000	5.0000
3-Nitroaniline	40.000	5.0000
4-Bromodiphenyl Ether	40.000	5.0000
4-Chloro-3-Methylphenol	40.000	5.0000
4-Chloroaniline	40.000	5.0000
4-Chlorodiphenyl Ether	40.000	5.0000
4-Nitroaniline	40.000	5.0000
4-Nitrophenol	40.000	5.0000
Acenaphthene	40.000	5.0000
Acenaphthylene	40.000	5.0000

Reviewed By: _____

Anthracene	40.000	5.0000
Azobenzene	40.000	5.0000
Benz(A)Anthracene	40.000	5.0000
Benzo(A)Pyrene	40.000	5.0000
Benzo(B)Fluoranthene	40.000	5.0000
Benzo(ghi)Perylene	40.000	5.0000
Benzo(K)Fluoranthene	40.000	5.0000
Benzyl Butyl Phthalate	40.000	5.0000
Bis(2-Chloroethoxy)Methane	40.000	5.0000
Bis(2-Chloroethyl)Ether	40.000	5.0000
Bis(2-Ethylhexyl)Phthalate	40.000	5.0000
Carbazole	40.000	5.0000
Chrysene	40.000	5.0000
Dibenz(A,H)Anthracene	40.000	5.0000
Dibenzofuran	40.000	5.0000
Dibutyl Phthalate	40.000	5.0000
Diethyl Phthalate	40.000	5.0000
Dimethyl Phthalate	40.000	5.0000
Dioctyl Phthalate	40.000	5.0000
Fluoranthene	40.000	5.0000
Fluorene	40.000	5.0000
Hexachloro-1,3-butadiene	40.000	5.0000
Hexachlorobenzene	40.000	5.0000
Hexachlorocyclopentadiene	40.000	5.0000
Hexachloroethane	40.000	5.0000
Indeno(1,2,3-cd)Pyrene	40.000	5.0000
Isophorone	40.000	5.0000
N-Nitrosodi-N-propylamine	40.000	5.0000
N-Nitrosodimethylamine	40.000	5.0000
Naphthalene	40.000	5.0000
Nitrobenzene	40.000	5.0000
O-Cresol	40.000	5.0000
P-Cresol	40.000	5.0000
Pentachlorophenol	40.000	5.0000
Phenanthrene	40.000	5.0000
Phenol	40.000	5.0000
Pyrene	40.000	5.0000

Reviewed By: _____

TestAmerica Pittsburgh

Standards Preparation Logbook Record

Sep-09-2009

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0893-09, +LLSSTD20(10ug/ml) Analyst: piccolinov

Solvent: Methylene chloride Lot No.: BNA0803-09 Volume (ml): 1.0000
Date Prep./Opened: 07-01-2009
Date Expires(1): 08-31-2009 (6 Months)
Date Reviewed: 08-18-2009 by bozike

Parent Std No.: BNA0854-08, 4,6dn2mphenol 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4,6-Dinitro-2-methylphenol	100.00	5.0000

Parent Std No.: BNA0855-08, 4 nitrophenol 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
4-Nitrophenol	20.000	1.0000

Parent Std No.: BNA0856-08, 2,4 dinitrophenol 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
2,4-Dinitrophenol	20.000	1.0000

Parent Std No.: BNA0857-08, benzoic acid 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Benzoic Acid	100.00	5.0000

Parent Std No.: BNA0858-08, pentachlorophenol 100ug/ml Aliquot Amount (ml): 0.0500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Pentachlorophenol	100.00	5.0000

Parent Std No.: BNA0866-09, LOW LEVEL Stock 40ug/ml Aliquot Amount (ml): 0.2500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Indene	40.000	10.000
2,4,6-Tribromophenol	40.000	10.000
2-Chlorophenol-d4	40.000	10.000
2-Fluorophenol	40.000	10.000
Phenol-d5	40.000	10.000
1,2-Dichlorobenzene-d4	40.000	10.000
2-Fluorobiphenyl	40.000	10.000
Nitrobenzene-d5	40.000	10.000
p-Terphenyl-d14	40.000	10.000

Reviewed By: _____

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Anthracene	40.000	10.000
Azobenzene	40.000	10.000
Benz(A)Anthracene	40.000	10.000
Benzo(A)Pyrene	40.000	10.000
Benzo(B)Fluoranthene	40.000	10.000
Benzo(ghi)Perylene	40.000	10.000
Benzo(K)Fluoranthene	40.000	10.000
Benzyl Butyl Phthalate	40.000	10.000
Bis(2-Chloroethoxy)Methane	40.000	10.000
Bis(2-Chloroethyl)Ether	40.000	10.000
Bis(2-Ethylhexyl)Phthalate	40.000	10.000
Carbazole	40.000	10.000
Chrysene	40.000	10.000
Dibenz(A,H)Anthracene	40.000	10.000
Dibenzofuran	40.000	10.000
Dibutyl Phthalate	40.000	10.000
Diethyl Phthalate	40.000	10.000
Dimethyl Phthalate	40.000	10.000
Dioctyl Phthalate	40.000	10.000
Fluoranthene	40.000	10.000
Fluorene	40.000	10.000
Hexachloro-1,3-butadiene	40.000	10.000
Hexachlorobenzene	40.000	10.000
Hexachlorocyclopentadiene	40.000	10.000
Hexachloroethane	40.000	10.000
Indeno(1,2,3-cd)Pyrene	40.000	10.000
Isophorone	40.000	10.000
N-Nitrosodi-N-propylamine	40.000	10.000
N-Nitrosodimethylamine	40.000	10.000
Naphthalene	40.000	10.000
Nitrobenzene	40.000	10.000
O-Cresol	40.000	10.000
P-Cresol	40.000	10.000
Pentachlorophenol	40.000	10.000
Phenanthrene	40.000	10.000
Phenol	40.000	10.000
Pyrene	40.000	10.000

Reviewed By: _____

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TestAmerica Pittsburgh

Standards Preparation Logbook Record Sep-09-2009

Controlled Source: Intranet

Logbook: \\pitsvr01\stdslog GCMS Semi-Volatile.std

BNA0894-09, +LLSSTD40(20ug/ml) Analyst: piccolinov

Solvent: Methylene chloride Lot No.: BNA0803-09 Volume (ml): 1.0000
Date Prep./Opened: 07-01-2009
Date Expires(1): 08-31-2009 (6 Months)
Date Reviewed: 08-18-2009 by bozike

Parent Std No.: BNA0866-09, LOW LEVEL Stock 40ug/ml Aliquot Amount (ml): 0.5000

1,2,4-Trichlorobenzene	40.000	20.000
1,2-Dichlorobenzene	40.000	20.000
1,3-Dichlorobenzene	40.000	20.000
1,4-Dichlorobenzene	40.000	20.000
2,4,5-Trichlorophenol	40.000	20.000
2,4,6-Trichlorophenol	40.000	20.000
2,4-Dichlorophenol	40.000	20.000
2,4-Dimethylphenol	40.000	20.000
2,4-Dinitrophenol	40.000	20.000
2,4-Dinitrotoluene	40.000	20.000
2,6-Dinitrotoluene	40.000	20.000
2-Chloroisopropyl Ether	40.000	20.000
2-Chloronaphthalene	40.000	20.000
2-Chlorophenol	40.000	20.000
2-Methyl-4,6-Dinitrophenol	40.000	20.000
2-Methylnaphthalene	40.000	20.000
2-Nitroaniline	40.000	20.000
2-Nitrophenol	40.000	20.000

Controlled Source: Intranet

Hexachloroethane	40.000	20.000
Indeno(1,2,3-cd)Pyrene	40.000	20.000
Isophorone	40.000	20.000
N-Nitrosodi-N-propylamine	40.000	20.000
N-Nitrosodimethylamine	40.000	20.000
Naphthalene	40.000	20.000
Nitrobenzene	40.000	20.000
O-Cresol	40.000	20.000
P-Cresol	40.000	20.000
Pentachlorophenol	40.000	20.000
Phenanthrene	40.000	20.000
Phenol	40.000	20.000
Pyrene	40.000	20.000

TestAmerica Pittsburgh

Standards Preparation Logbook Record

Sep-09-2009

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0895-09, +LLSSTD60(30ug/ml) Analyst: piccolinov

Solvent: Methylene chloride Lot No.: BNA0803-09 Volume (ml): 1.0000
Date Prep./Opened: 07-01-2009
Date Expires(1): 08-31-2009 (6 Months)
Date Reviewed: 08-18-2009 by kozike

Controlled Source: Intranet

Parent Std No.: BNA0866-09, LOW LEVEL Stock 40ug/ml Aliquot Amount (ml): 0.7500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Indene	40.000	30.000
2,4,6-Tribromophenol	40.000	30.000
2-Chlorophenol-d4	40.000	30.000
2-Fluorophenol	40.000	30.000
Phenol-d5	40.000	30.000

1,2,4-Trichlorobenzene	40.000	30.000
1,2-Dichlorobenzene	40.000	30.000
1,3-Dichlorobenzene	40.000	30.000
1,4-Dichlorobenzene	40.000	30.000
2,4,5-Trichlorophenol	40.000	30.000
2,4,6-Trichlorophenol	40.000	30.000
2,4-Dichlorophenol	40.000	30.000
2,4-Dimethylphenol	40.000	30.000
2,4-Dinitrophenol	40.000	30.000
2,4-Dinitrotoluene	40.000	30.000
2,6-Dinitrotoluene	40.000	30.000
2-Chloroisopropyl Ether	40.000	30.000
2-Chloronaphthalene	40.000	30.000
2-Chlorophenol	40.000	30.000
2-Methyl-4,6-Dinitrophenol	40.000	30.000
2-Methylnaphthalene	40.000	30.000
2-Nitroaniline	40.000	30.000
2-Nitrophenol	40.000	30.000
3-Nitroaniline	40.000	30.000
4-Bromodiphenyl Ether	40.000	30.000
4-Chloro-3-Methylphenol	40.000	30.000
4-Chloroaniline	40.000	30.000
4-Chlorodiphenyl Ether	40.000	30.000
4-Nitroaniline	40.000	30.000
4-Nitrophenol	40.000	30.000
Acenaphthene	40.000	30.000
Acenaphthylene	40.000	30.000

Hexachloroethane	40.000	30.000
Indeno(1,2,3-cd)Pyrene	40.000	30.000
Isophorone	40.000	30.000
N-Nitrosodi-N-propylamine	40.000	30.000
N-Nitrosodimethylamine	40.000	30.000
Naphthalene	40.000	30.000
Nitrobenzene	40.000	30.000
O-Cresol	40.000	30.000
P-Cresol	40.000	30.000
Pentachlorophenol	40.000	30.000
Phenanthrene	40.000	30.000
Phenol	40.000	30.000
Pyrene	40.000	30.000

TestAmerica Pittsburgh

Standards Preparation Logbook Record

Sep-09-2009

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0896-09, +LLSSTD80(40ug/ml) Analyst: piccolimov

Solvent: Methylene chloride Lot No.: BNA0803-09 Volume (ml): 1.0000
Date Prep./Opened: 07-01-2009
Date Expires(1): 08-31-2009 (6 Months)
Date Reviewed: 08-18-2009 by bozike

Parent Std No.: BNA0866-09, LOW LEVEL Stock 40ug/ml Aliquot Amount (ml): 1.0000

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitroso-di-n-butylamine	40.000	40.000
1,4 Dioxane	40.000	40.000
1,2,4,5-Tetrachlorobenzene	40.000	40.000
Acetophenone	40.000	40.000
Atrazine	40.000	40.000
Benzaldehyde	40.000	40.000
Biphenyl	40.000	40.000
Caprolactam	40.000	40.000
1-Nitrosopyrrolidine	40.000	40.000
Aniline	40.000	40.000
Benzoic Acid	40.000	40.000
Benzyl Alcohol	40.000	40.000
Pyridine	40.000	40.000
N-Nitrosodiphenylamine	40.000	40.000
2,6-Dichlorophenol	40.000	40.000
1,2,4-Trichlorobenzene	40.000	40.000
1,2-Dichlorobenzene	40.000	40.000

3-Nitroaniline	40.000	40.000
4-Bromodiphenyl Ether	40.000	40.000
4-Chloro-3-Methylphenol	40.000	40.000
4-Chloroaniline	40.000	40.000
4-Chlorodiphenyl Ether	40.000	40.000
4-Nitroaniline	40.000	40.000
4-Nitrophenol	40.000	40.000
Acenaphthene	40.000	40.000
Acenaphthylene	40.000	40.000
Anthracene	40.000	40.000
Azobenzene	40.000	40.000
Benz(A)Anthracene	40.000	40.000
Benzo(A)Pyrene	40.000	40.000
Benzo(B)Fluoranthene	40.000	40.000
Benzo(ghi)Perylene	40.000	40.000
Benzo(K)Fluoranthene	40.000	40.000
Benzyl Butyl Phthalate	40.000	40.000
Bis(2-Chloroethoxy)Methane	40.000	40.000
Bis(2-Chloroethyl)Ether	40.000	40.000
Bis(2-Ethylhexyl)Phthalate	40.000	40.000
Carbazole	40.000	40.000
Chrysene	40.000	40.000
Dibenz(A,H)Anthracene	40.000	40.000
Dibenzofuran	40.000	40.000
Dibutyl Phthalate	40.000	40.000
Diethyl Phthalate	40.000	40.000
Dimethyl Phthalate	40.000	40.000
Dioctyl Phthalate	40.000	40.000
Fluoranthene	40.000	40.000
Fluorene	40.000	40.000
Hexachloro-1,3-butadiene	40.000	40.000
Hexachlorobenzene	40.000	40.000
Indene	40.000	40.000
2,4,6-Tribromophenol	40.000	40.000
2-Chlorophenol-d4	40.000	40.000
2-Fluorophenol	40.000	40.000
Phenol-d5	40.000	40.000

Hexachloroethane	40.000	40.000
Indeno(1,2,3-cd)Pyrene	40.000	40.000
Isophorone	40.000	40.000
N-Nitrosodi-N-propylamine	40.000	40.000
N-Nitrosodimethylamine	40.000	40.000
Naphthalene	40.000	40.000
Nitrobenzene	40.000	40.000
O-Cresol	40.000	40.000
P-Cresol	40.000	40.000
Pentachlorophenol	40.000	40.000
Phenanthrene	40.000	40.000
Phenol	40.000	40.000
Pyrene	40.000	40.000

TestAmerica Pittsburgh

Standards Preparation Logbook Record

Sep-09-2009

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0817-09, LL2nd source verif sstd20(10ug/ml) Analyst: piccolinov

Solvent: Methylene chloride Lot No.: bna0803-09 Volume (ml): 1.0000
Date Prep./Opened: 06-06-2009
Date Expires(1): 07-31-2009 (6 Months)

Parent Std No.: BNA0233-09, Indene Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 01-12-2010 Parent Date Expires(2): 09-29-2010

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Indene	1,000.0	10.000

Parent Std No.: BNA0568-09, Equity SS 8270 Benzidines Mix Aliquot Amount (ml): 0.0050
Parent Date Expires(1): 03-20-2010 Parent Date Expires(2): 08-31-2010

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
3,3'-Dichlorobenzidine	2,000.0	10.000
3,3'-Dimethylbenzidine	2,000.0	10.000
Benzidine	2,000.0	10.000

Parent Std No.: BNA0569-09, Equity SS N-Nitrosodiphenylamine Aliquot Amount (ml): 0.0020
Parent Date Expires(1): 07-31-2009 Parent Date Expires(2): 07-31-2009

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosodiphenylamine	5,000.0	10.000

Parent Std No.: BNA0570-09, Equity SS 8270 Calibration Mix Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 03-20-2010 Parent Date Expires(2): 11-30-2010

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
64 Compounds	1,000.0	10.000

Controlled Source: Intranet

Parent Std No.: BNA0571-09, Equity SS 8270 Calibration Mix 4 Aliquot Amount (ml): 0.0050
Parent Date Expires(1): 07-31-2009 Parent Date Expires(2): 07-31-2009

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Aniline	2,000.0	10.000
Benzoic Acid	2,000.0	10.000
Benzyl Alcohol	2,000.0	10.000
Pyridine	2,000.0	10.000

Parent Std No.: BNA0572-09, 2,6 Dichlorophenol Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 03-20-2010 Parent Date Expires(2): 06-20-2012

7-compounds	2,000.0	10.000

Parent Std No.: BNA0656-09, 1,4 Dioxane 2nd source — Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 04-21-2010 Parent Date Expires(2): 02-25-2014

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,4 Dioxane	1,000.0	10.000

Parent Std No.: BNA0695-09, 1,2,4,5-Tetrachlorobenzene 2nd source — Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 05-02-2010 Parent Date Expires(2): 09-09-2013

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,2,4,5-Tetrachlorobenzene	1,000.0	10.000

Parent Std No.: BNA0752-09, Equity SS CLP OLM04 Semivolatiles — Aliquot Amount (ml): 0.0050
Parent Date Expires(1): 05-21-2010 Parent Date Expires(2): 03-31-2011

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Acetophenone	2,000.0	10.000
Atrazine	2,000.0	10.000
Benzaldehyde	2,000.0	10.000
Biphenyl	2,000.0	10.000
Caprolactam	2,000.0	10.000

Parent Std No.: BNA0816-09, N-Nitrosopyrrolidine 2nd source — Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 06-06-2010 Parent Date Expires(2): 06-11-2014

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosopyrrolidine	1,000.0	10.000

Appendix B– EPA Memo Regarding Method 625 Modification

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

MEMORANDUM

OFFICE OF WATER

SUBJECT: **Recommended Approved Modifications to EPA Method 625**

FROM: Richard Reding, Chief
Engineering & Analytical Support Branch, EAD, OST

TO: Quality Assurance Managers
ATP Coordinators
NPDES Coordinators

DATE: November 1, 2006

The 304(h) methods branch recommends allowing several modifications to EPA Method 625 for environmental permitting and compliance monitoring under the EPA's Clean Water Act (CWA) programs. This memorandum does not address laboratory certification requirements that states have mandated.

The text in "Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water" Section 1.3.2 allows flexibility in the modification of "front end techniques" of the test method provided all criteria in this section and **all QC in the method are** met and documented. This protocol can be downloaded at http://www.epa.gov/waterscience/methods.

Recommendations on Method Modifications to EPA Method 625 when Capillary Columns are used:

1. **Combining sample extracts before analysis**

If the analytes can be reliably identified and quantified in the combined extracts, the extracts may be combined. If, however, the identification and quantitation of any analyte is adversely affected by another analyte, a surrogate, or an interferant, the extracts must be analyzed separately. If there is ambiguity, the extracts must be analyzed separately.

2. **Reverse order of pH extraction**

The pH extraction sequence may be reversed to better separate acid and neutral components. Neutral components may be extracted with either acid or base components.

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Previously, neither of these modifications has been used with Method 625 primarily because of limitations of the resolving power of the packed columns used. In 1985, EPA Region 3 Central Regional Lab requested a modification to method 625 as an alternate test procedure (ATP). Although the approval was for limit use by EPA's Region 3, Central Regional Laboratory only, this modification has come to be used throughout the laboratory community (see attached memo).

Why allow these modifications? Following the base-neutral than acid extraction sequence of method 625 in some cases demonstrated the decomposition of some analytes under basic conditions. Organochlorine pesticides may dechlorinate; phthalate esters may exchange; phenols may react to form tannates. These reactions increase with increasing pH. Reversing the extraction pH sequence may better separate acid and neutral waste components.

Other Recommended Modifications to Method 625

A smaller sample volume may be used to minimize matrix interferences provided matrix interferences are demonstrated and documented.

Alternate surrogate and internal standard concentrations other than those specified in the method are acceptable provided that method performance is not degraded;

An alternate calibration curve and a calibration check other than those specified in the method;

A different solvent for the calibration standards to match the solvent of the final extract.

Other Method Flexibility News

We are revising the "Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring" often referred to as the "Pumpkin Book". Many of the recommendations in the revised "Pumpkin Book" cover ways to mitigate matrix effects.

More explicit flexibility to make changes in approved methods without prior EPA approval is now described at 40 CFR Part 136.6. Such changes are only allowed if the modified method produces equivalent performance for the analyte(s) of interest, and the equivalent performance is documented. It is essential to consult the full text at 40 CFR 136.6 before undertaking method modifications.

Please feel free to forward this information. If you have any questions regarding this memorandum, please contact Lemuel Walker of EASB/EAD/OST by email at walker.lemuel@epa.gov.

cc Lemuel Walker
ATP Coordinator

Appenidx C

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081A: Endrin, DDT 8270C: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.
CCV	This verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.	To verify that instrument response is reliable, and has not changed significantly from the current ICAL.	If the values for the analytes are outside the acceptance criteria, the initial calibration may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging.
Demonstrate Acceptable Analyst Capability	Analyst runs QC samples in series to establish his/her ability to produce data of acceptable accuracy and precision.	To establish the analysts' ability to produce data of acceptable accuracy and precision.	The average recovery and standard deviation of the replicate must be within designated acceptance criteria.
Duplicate Sample	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are taken and prepared and analyzed identically.	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix.	To provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the sample and the duplicate.
ICAL	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method.	To establish a calibration curve for the quantification of the analytes of interest.	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.

Appenidx C

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Internal Standards	A known amount of standard added to all standards and samples as a reference for evaluating and controlling the precision and bias of the applied analytical method.	To verify that the analytical system is in control.	Any sample associated with out-of-control results must be reanalyzed.
LCS containing all analytes required to be reported	A QC standard of known composition prepared using reagent free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern.	To evaluate method performance by assessing the ability of the lab/analyst to successfully recover the target analytes from a control (clean) matrix.	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the accuracy/bias of the measurement system.
MS	A sample prepared by adding a know amount of targeted analyte(s) to an aliquot of a specific environmental sample.	To assess the performance of the method as applied to a particular matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty.
MSD	A 2nd replicate MS prepared in the lab, spiked with an identical, known amount of targeted analyte(s), and analyzed to obtain a measure of the precision of the recovery for each analyte.	To assess the performance of the method as applied to a particular matrix and provide information of the homogeneity of the matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix.
MDL Verification Check	A low-level spike taken through the prep and analytical steps at approximately 2x the MDL used to verify that the laboratory can detect analytes at the calculated MDL.	To validate the MDL on an ongoing basis	If the MDL verification check fails, reprep/reanalyze at a higher level to set a higher MDL or the MDL study must be repeated.
MB	A sample of a matrix similar to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical results.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data.	This QC is used to measure lab accuracy/bias. The MB could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or B-Flag results for all samples in prep batch. For common

Appenidx C

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
			lab contaminants, no analytes detected > RL. See DoD Box D-5; & Sec. D.1.1.1
MDL Study	The process to determine the minimum concentration of an 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 containing the analyte.	To determine the lowest concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.	MDLs must be established prior to sample analysis. The RL or LOQ is at least 3x the MDL. Used in combination with the MDL verification check to validate the MDL on an ongoing basis.
RT window position establishment for each analyte (chromatographic methods only)	Determination of the placement of the RT window (start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the initial calibration.	To identify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, and/or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
RT window verification for each analyte (chromatographic methods only)	A standard is used to verify that the width and position of the RT windows are valid so that accurate analyte identification can be made during sample analysis.	To minimize the occurrence of both false positive and false negative results at each calibration verification.	The peaks from the standard used are compared to the RT window established during the ICAL to verify that the analytes of interest still fall within the window.
Second source calibration verification	A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.	To verify the accuracy of the initial calibration.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A pure substance with properties that mimic the analyte of interest. Surrogates are compounds unlikely to be found in environmental samples to evaluate analytical efficiency by measuring their % Recovery.	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix.	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS; MS), the bias in the analytical system can be determined.

Appenidx C

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Tuning (MS methods only)	The analysis of a standard compound to verify the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis.	To verify the proper working of the mass spectrometer.	Proper tuning of the mass spectrometer must be verified prior to sample analysis .

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.

2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
IDOC	Per Instrument/Analyst	DoD acceptance criteria if available; otherwise method specific criteria.	Correct / Repeat for those analytes which failed criteria.	NA
MDL	Annually or quarterly MDL Checks performed	40 CFR 136B; MDL verification checks must produce a signal at least 3x the instrument's noise level.	Run MDL check at higher level and set MDL higher or reconduct MDL study.	NA
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method specific ion criteria.	Retune instrument and verify. Rerun affected samples.	NA
Breakdown check DDT (8270C only)	Daily prior to analysis of samples	Degradation < 20% for DDT (Benzidine & PCP should be present at their normal response and no peak tailing should be observed).	Correct problem then repeat breakdown check.	NA
ICAL	Initial 5-point calibration prior to sample analysis	1. Average RF for SPCCs: SVOCs - > 0.050 2. RSD for RFs for CCCs: SVOCs - < 30% and one option below. **Option 1:** RSD for each analyte < 15% **Option 2:** linear least squares regression: r > 0.995 **Option 3:** non-linear regression: Coefficient of determination (COD) r^2 > 0.99 (6 points shall be used for 2nd order, 7 points shall be used for 3rd order)	Correct problem then repeat initial calibration.	NA
2nd Source calibration verification	Once after each initial calibration	Value of 2nd source for all analytes within + 25% of expected value - See SOP Section 10.1.10 for exception and DoD SOP.	Correct problem and verify 2nd source standard. Rerun, if that fails, correct problem and repeat ICAL.	NA
RT window position	Once per ICAL	Position shall be set using midpoint standard of the initial calibration	NA	NA

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
establishment for each analyte		curve.		
Evaluation of Relative RT (RRT)	With each sample	RRT of each target analyte in each calibration standard within + 0.06 RRT units.	Correct problem, then rerun ICAL	NA
Calibration verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	1. Average RF for SPCCs: SVOCs - > 0.050 2. %Difference for CCCs: SVOCs - < 20% D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration) All calibration analytes must be within 20% D, with no individual analytes (except CCC's) > 25% D	Correct problem, rerun CV, if fails, repeat ICAL (Data associated with an unacceptable CCV may be fully usable under the following conditions: 1. CCV (high bias) and samples ND, then raw data may be reported with appropriate flag 2. CCV (low bias) and samples exceed maximum regulatory limit/decision level (DoD Box 60: Project specific permission from appropriate DoD personnel is required to report data generated from a run with noncompliant CCV.)	Apply J-flag to all results associated with the analytical batch for all analytes > 20%D and < 25% D. Identify in case narrative analytes > 20% D.) Apply Q-flag if no sample material remains and analyte exceeds criteria
Internal Standards verification	In all field samples and standards	RT + 30 seconds from RT of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Apply Q-flag to analytes associated with the non-compliant IS.
MB	One per prep batch	No analytes detected > ½ RL For common lab contaminants, no analytes > RL	Correct problem, then see criteria in box D-5; if required, reprep/reanalyze MB and all associated samples.	Apply B-flag to all results for the contaminated analyte for all samples in the associated prep batch.
LCS (containing all analytes to be reported)	One LCS per prep batch	DoD specified QC criteria, if available	Correct problem, reprep/reanalyze the LCS and all samples in the associated prep batch for all failed analytes, if sufficient sample is available.	Apply Q-flag to specific analyte(s) in all samples in the prep batch.

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
			available.	
MS	One per prep batch per matrix	For matrix evaluation, use DoD specified QC criteria for LCS.	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample.
MSD or Sample Duplicate	One per prep batch per matrix	RPD < 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample.
Surrogate	All field and QC samples	DoD specified QC criteria if available, otherwise method specific criteria or lab's own in-house criteria.	For QC and field samples, correct problem, reprep/reanalyze all failed samples in the associated prep batch if sufficient sample material is available.	Apply J-flag for specific analyte(s) in all field samples collected from the same site matrix as the parent. Apply Q-flag to QC samples for specific analyte(s)
Results reported between LOD and LOQ			Apply J-flag to all results between LOD (MDL) and LOQ (RL)	
Manual Integration	When manual integrations are performed	Raw data shall include a complete audit trail for those manipulations, raw data output showing the results of the MI (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and signature/initials of person performing manual operation.		Apply M-flag to MI data

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.

2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

Title: Chlorinated Pesticides

Method: SW-846 8081B

Approvals (Signature/Date):

Sharon Bacha Technical Manager	7/14/2010 Date	Steve Jackson Health & Safety Manager/Coordinator	6/30/2010 Date
Nasreen DeRubeis Quality Assurance Manager	07/15/10 Date	Larry Matko Laboratory Director	6/29/2010 Date

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1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of chlorinated pesticides using the methodology described in EPA SW-846 Method 8081B.

1.2 This SOP is applicable to the gas chromatographic (GC) analysis of extracts of soil, sediment, tissue, oil, waste and water samples. Table 1 lists the compounds that can be determined by this method and their associated routine reporting limits (RLs).

1.3 This SOP does not include the procedures for extracting soil and water samples. Refer to SOP PT-OP-001 for sample extraction procedures. For specific DoD quality control requirements refer to SOP # PT-QA-025, Implementation of the DoD QSM Version 3, January 2006 and Appendix I in this SOP. For DoD V4.1 refer to SOP PT-QA-029.

1.4 Analytes, Matrix(s), and Reporting Limits: See Table 1 for analytes and reporting limits by matrix.

2.0 Summary of Method

2.1 Sample Preparation

2.1.1 Chlorinated pesticides are typically extracted from a one-liter water sample with methylene chloride using a separatory funnel (Method 3510C). Detailed instructions are given in SOP PT-OP-001. The methylene chloride extract is exchanged to hexane as described in SOP PT-OP-001.

2.1.2 Chlorinated pesticides are typically extracted from a 15-gram soil/tissue/sediment subsample into a 50:50 acetone-methylene chloride solution by automated soxtherm (Method 3541). The extract is dried and exchanged to hexane. Detailed instructions are given in SOPs PT-OP-001.

2.1.3 SOP PT-OP-001 provides instructions for the concentration and cleanup of sample extracts. Carboprep 90 Cleanup is used to clean extracts that show color. Sulfur is removed if observed. All extracts are in hexane and the final extract volume is 20 mL for soils and 40 mL for waters.

2.2 Analysis

2.2.1 Samples are analyzed using a gas chromatograph equipped with dual columns and dual electron capture detectors (ECDs).

2.2.2 The instrument is calibrated using external standards. Compounds are identified by their retention time on the columns.

2.2.3 Positive results from the primary column are confirmed with a second, dissimilar column.

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3.0 Definitions

3.1 Single-Component Pesticides: A pesticide formulation that consists of a single chemical compound. Most of the analytes determined by this procedure are single-compound pesticides.

3.2 Multi-Component Pesticides: A pesticide formulation that consists of more than one chemical compound. Toxaphene and Technical Chlordane are production mixtures of multiple compounds. Toxaphene is manufactured by the chlorination of camphenes, which produces a variety of compounds, not all of which are chromatographically resolved. Technical Chlordane is produced by the chlorination of a mixture of camphenes and pinenes.

3.3 Chlordane: As just described, Technical Chlordane (CAS# 12789-03-6) is a mixture of compounds. Method 8081B, Section 11.6.2 notes that it includes at least 11 major components and 30 minor components, and adds “the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch.” The laboratory has found that manufacturing lots of Technical Chlordane produced at different times or at different production facilities have different ratios of the key components. For this reason, it is more common to analyze for the major components of technical Chlordane (α-Chlordane, γ-Chlordane, and heptachlor) instead of analyzing for the total mixture. For the purpose of reporting results under this SOP, the following compounds are reported. Alpha-chlordane (cis-chlordane) CAS # 5103-71-9 and gamma-chlordane (trans-chlordane) CAS # 5103-74-2. The laboratory may also report chlordane (not otherwise specified) or, n.o.s under CAS# 57-74-9.

3.4 The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Pittsburgh Laboratory Quality Assurance Manual (PT-LQAM).

4.0 Interferences

4.1 Contamination by carryover can occur when a low concentration sample is analyzed immediately following a high concentration sample. It is the laboratory's policy to evaluate and reanalyze when necessary any samples that follow an unusually concentrated sample and that show detectable levels of the same compounds that appeared in the preceding concentrated sample.

4.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3 Sulfur will interfere, and, when observed, is removed using cleanup procedures described in SOP PT-OP-001.

4.4 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), Carboprep 90 Cleanup and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # PT-OP-001. Use of hexane / acetone as the extraction solvent (rather than acetone / methylene chloride) may reduce the amount of interferences extracted.

4.5 Carboprep 90 Cleanup

4.5.1 This cleanup may be performed prior to analyses for PCBs if the sample extract has some color.

4.5.2 Cartridge Method

4.5.2.1 Put approximately 2 ml of sample extract into a test tube and mark the sample volume on the tube.

4.5.2.2 Condition the cartridge by adding 2 ml of methylene chloride and allowing it to drip through the cartridge. Do not allow the cartridge packing to go dry in this or any subsequent step, until the final rinse has been completed.

4.5.2.3 Add 2 ml of hexane/methylene chloride (80%/20%) mixture and allow it to drip through the cartridge until almost empty.

4.5.2.4 Add the sample extract to the cartridge and place the test tube under the cartridge to collect the liquid as it drips through.

4.5.2.5 Rinse 3 times with 2 ml aliquots of hexane/methylene chloride (80%/20%) mixture, while not allowing the cartridge to go dry. After the final rinse, use a pipette bulb to force out all of the remaining liquid in the cartridge.

4.5.2.6 Concentrate the sample extract back down to the original volume according to the mark on the test tube. The extract is now ready for analysis.

4.5.3 Quick Method

4.5.3.1 Add a half scoop of Carboprep 90 to approximately 2 ml of sample extract. Shake for one minute and allow the extract to settle. Pipette out an aliquot of the clear extract for analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 The ECD contains a ^{63}Ni radioactive source. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the facility's radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Radiation Safety Officer shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment.

5.1.5 As a safety precaution, all standards, samples, and extracts are handled in an approved fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects are exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6 Equipment and Supplies

6.1 An analytical system complete with a gas chromatograph and dual ECD (Ni-63) detectors is required. A data system capable of measuring peak area and/or height is required.

6.2 An analytical balance capable of weighing to 0.0100g.

6.3 Columns

6.3.1 Primary Column: MR1, 30 m X 0.53 mm id

6.3.2 Secondary Column: MR2, 30 m X 0.53 mm id

6.3.3 Additional columns that can be used for confirmation include 30 m X 0.53 mm id RTx5, RTx50 or RTx1701.

6.4 Autosampler vials, crimp-top cap with PTFE-faced septa

6.5 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7 Reagents and Standards

7.1 Reagents

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7.1.1 Hexane, pesticide grade

7.1.2 Carrier gas, ≥ 99.99999% pure hydrogen or helium

7.1.3 Make-up gas, ≥ 99.99980% pure nitrogen

7.2 Standards

7.2.1 Storage of Stock Standards

7.2.1.1 Commercial standards are received in flame-sealed ampoules or neat, 100% concentration, solutions. Stock standards are stored refrigerated at ≤ 6 ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

7.2.1.2 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem. Kepone extracted from samples or in standards exposed to water or methanol may produce peaks with broad tails that elute later than the standard by up to 1 minute. This shift is presumably the result of the formation of a hemi-acetal from the ketone functionality and may seriously affect the ability to identify this compound on the basis of retention time. For this reason, analysis for Kepone using Method 8081B is not performed by TestAmerica Pittsburgh. Method 8270D is the method of choice for this compound. Endosulfan I and II appear to degrade in the presence of methanol. Gamma-BHC appears to degrade in the presence of acetone.

7.3 Calibration Stock Standards

7.3.1 All calibration stock standards are obtained from commercial sources.

NOTE: The availability of the specific commercial standard solutions upon which the following sections are based may change at any time. As a result, it may be necessary to alter the dilution scheme presented herein to accommodate changes in stock standard concentrations. All such changes are documented in the standards preparation records.

7.3.2 Routine Pesticide A and B Mix Stock Standard

Custom Pesticide Mix A	
Compound	**Concentration (µg/mL)**
TCMX (surrogate)	100
4,4'-DDD	100
4,4'-DDT	100

Custom Pesticide Mix A	
Compound	**Concentration (µg/mL)**
α-BHC	100
DCB (surrogate)	100
Dieldrin	100
Endosulfan I	100
Endrin	100
γ-BHC (Lindane)	100
Heptachlor	100
Methoxychlor	200
Custom Pesticide Mix B	
4,4'DDE	100
Aldrin	100
α-Chlordane	100
β-BHC	100
δ-BHC	100
Endosulfan II	100
Endosulfan Sulfate	100
Endrin aldehyde	100
Endrin ketone	100
γ-Chlordane	100
Heptachlor epoxide	100

7.3.3 Surrogate Stock Standard

7.3.3.1 The surrogate stock standard contains decachlorobiphenyl (DCB) at 1000 µg/mL and tetrachloro-m-xylene (TCMX) is contained in a separate stock standard at 2000 µg/mL.

7.3.4 Toxaphene Stock, 1000 µg/mL

7.3.4.1 The Toxaphene stock standard contains a specific production mixture of Toxaphene. This mixture does not necessarily match all possible production mixtures that could be found in the environment. This can present problems for Toxaphene quantitation (see Section 12).

7.3.5 Chlordane Stock, 100 µg/mL

7.3.5.1 The Toxaphene stock standard contains a specific production mixture of Toxaphene. This mixture does not necessarily match all possible production mixtures that could be found in the environment. This can present problems for Toxaphene quantitation (see Section 12).

7.3.5.2 The Chlordane stock contains Technical Chlordane (CAS# 12789-03-6).

7.3.6 Appendix IX Calibration Stock

7.3.6.1 The Appendix IX stock calibration mixture contains the compounds at the concentrations listed in the following table.

Appendix IX Mix 1 and Mix 2 Calibration Stock Standard

Mix 1 Compounds	Concentration (µg/mL)
Chlorobenside	50
Dacthal (DCPA)	50
Hexachlorobenzene	25
Hexachlorocyclopentadiene	50
Mirex	20
Isodrin	10
Oxychlordane isomer	10
Mix 2 Compounds	**Concentration (µg/mL)**
2,4-DDD	10
2,4-DDE	10
2,4-DDT	10
Chlorobenzilate	100
cis-Nonachlor	10
DCB (surrogate)	10
Diallate	200
Hexachlorobutadiene	10
TCMX (surrogate)	10
trans-Nonachlor	10

7.3.7 Non-Routine Compounds

7.3.7.1 Other, non-routine compounds not listed in this section may be requested by a client and may be added to this procedure.

7.3.7.2 In these cases, all stock solutions will be obtained from commercial sources and will be verified with a second-source standard as described in Section 7.5.

7.3.7.3 Non-routine standards will be stored and treated as described in Section 7.2.1.

7.3.7.4 Subsequent dilutions of specially requested compounds will be determined in a manner

consistent with the client's recommendations for number of calibration points, inclusion of reporting limit, and concentration range adequate to represent the linearity of the instrument.

7.3.7.5 These specially requested, non-routine compounds either may be added to the dilution scheme used for routine compounds or may be prepared as a separate calibration.

7.3.7.6 All standards preparation for non-routine compounds shall be documented using the same method that is used for routine compounds.

7.4 Working Level Calibration Standards

7.4.1 The single peak pesticide stock standards are purchased as certified standards in two separate solutions in 50%hexane/50% toluene, which are combined prior to standard preparation (See Section 7.3.2). An intermediate stock standard is prepared by diluting 1.0 mL of each of the appropriate stock mixtures (Custom Pesticide Mix A and B) to 10.0 mL in hexane. The intermediate stock mix concentrations are 10 ug/mL for all compounds except methoxychlor, which is 20 ug/mL. The working standards are prepared by diluting the volume noted in the Table listed below to a 40.0 mL final volume in hexane except for the Level 3 standard, which is taken to a 100 mL final volume in hexane. (See Table below.)

7.4.2 Toxaphene and Technical Chlordane stock standards are purchased certified solutions at 100 ug/mL. The mid level (Level 3) Toxaphene calibration standard is prepared by diluting 0.40 mL of the stock standard mix to 40 mL in hexane. The mid level (Level 3) Technical Chlordane calibration standard is prepared by diluting 0.10 mL of the stock standard mix to 40 mL in hexane. (See Table below.)

Preparation of Calibration Standards						
	Calibration Level (µg/mL)[1]					
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**
Aldrin	0.001	0.005	0.025	0.050	0.100	0.200
α-BHC	0.001	0.005	0.025	0.050	0.100	0.200
β-BHC	0.001	0.005	0.025	0.050	0.100	0.200
δ-BHC	0.001	0.005	0.025	0.050	0.100	0.200
γ-BHC (Lindane)	0.001	0.005	0.025	0.050	0.100	0.200
4,4'-DDD	0.001	0.005	0.025	0.050	0.100	0.200
4,4'-DDE	0.001	0.005	0.025	0.050	0.100	0.200
4,4'-DDT	0.001	0.005	0.025	0.050	0.100	0.200
Dieldrin	0.001	0.005	0.025	0.050	0.100	0.200
Endosulfan I	0.001	0.005	0.025	0.050	0.100	0.200
Endosulfan II	0.001	0.005	0.025	0.050	0.100	0.200
Endosulfan Sulfate	0.001	0.005	0.025	0.050	0.100	0.200

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Preparation of Calibration Standards						
	Calibration Level (µg/mL)[1]					
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**
Endrin	0.001	0.005	0.025	0.050	0.100	0.200
Endrin Aldehyde	0.001	0.005	0.025	0.050	0.100	0.200
Endrin ketone	0.001	0.005	0.025	0.050	0.100	0.200
Heptachlor	0.001	0.005	0.025	0.050	0.100	0.200
Heptachlor Epoxide	0.001	0.005	0.025	0.050	0.100	0.200
Methoxychlor	0.002	0.010	0.050	0.100	0.200	0.400
Toxaphene			1.0			
Chlordane (tech.)			0.250			

[1]These calibration levels are prepared by adding 4, 20, 250, 200, 400 and 800 µL of the stock standard concentrations listed above. The mid-level Toxaphene and Chlordane calibration levels are prepared by adding 40 µL and 100 µL, respectively. **NOTE:** These amounts are subject to change as the concentration of the purchased standards changes.

7.4.3 Appendix IX Working Level Calibration Standards

7.4.3.1 The following volumes of the Appendix IX intermediate stock standard are diluted with hexane to a final volume of 25.0 mL. The following table summarizes the final compound concentration ranges for each calibration level. The concentration for each compound at each level is given in Table 3.

Appendix IX Mix 1 and 2 Working Level Calibration Standards		
Level	**Volume of Stock Std (mL)**	**Final Compound Concentration Range (µg/mL)**
1	0.005	0.0004 - 0.002
2	0.025	0.002 – 0.01
3*	0.20	0.05 – 0.25
4	0.25	0.02 – 0.10
5	0.50	0.04 – 0.20
6	1.0	0.08 – 0.40
* *This level is used as the CCV.*		

7.5 Second-Source Standards for Initial Calibration Verification (ICV)

7.5.1 The second-source stock standards are purchased from a vendor different from the one that supplied the stock calibration standards.

7.5.2 Routine Pesticide AB Mix ICV Stock Standard, 2,000 µg/mL

7.5.2.1 Commercial standards containing all single-component pesticide compounds are obtained from a vendor different from the one that supplied the calibration stock standard. Typically, the standards are obtained from Restek.

7.5.3 Appendix IX ICV Stock Standard

7.5.3.1 Commercial standards are obtained at the same concentrations as shown for the calibration stock standards in Section 7.3, but from a different vendor (typically AccuStandard).

7.5.4 Routine Pesticide ICV Working Level Standard, 0.025 µg/mL

7.5.4.1 The working level ICV standard for the routine pesticide compounds is prepared by diluting the ICV intermediate standard (Section 7.4) in hexane follows:

Routine Pesticide Second-Source ICV Working Level Standard		
Volume of Intermediate Standard (mL)	**Final Volume (mL)**	**Final Concentration (µg/mL)**
0.100	100	0.025

7.5.5 Appendix IX ICV Working Level Standard

7.5.5.1 The working level ICV standard for the Appendix IX compounds is prepared by diluting 0.040 mL of the second-source Appendix IX stock standard (Section 7.5.3) with hexane to a final volume of 40 mL. The following table lists the final concentration of each pesticide:

Appendix IX Second Source ICV Working Level Standard	
Mix 1	**Final Concentration (µg/mL)**
Chlorobenside	0.05
Dacthal (DCPA)	0.05
Hexachlorobenzene	0.025
Hexachlorocyclopentadiene	0.05

Appendix IX Second Source ICV Working Level Standard	
Mix 1	**Final Concentration (µg/mL)**
Mirex	0.01
Isodrin	0.02
Oxychlordane isomer	0.01
Mix 2	**Final Concentration (µg/mL)**
2,4-DDD	0.025
2,4-DDE	0.025
2,4-DDT	0.025
Chlorobenzilate	0.250
cis-Nonachlor	0.025
DCB (surrogate)	0.500
Diallate	0.025
Hexachlorobutadiene	0.025
TCMX (surrogate)	0.025
trans-Nonachlor	0.025

7.6 Continuing Calibration Verification (CCV) Standards

7.6.1 The Level 3 AB mix working calibration standard (Section 7.4.1 and 7.4.2) and the Level 3 Appendix IX working calibration standard (Section 7.5.5) are used as the CCV standard.

7.7 RL Standard

7.7.1 The lowest concentration calibration standard (i.e., Level 1) is used as the RL standard.

7.8 Laboratory Control Standard (LCS) Spike Solution, 1.0 µg/mL

7.8.1 The working LCS spike solution is prepared by diluting 0.100 mL of a commercially purchased (2000 µg/mL) AB mix stock standard in acetone to a final volume of 200 mL in a volumetric flask, as summarized in the table below.

7.8.2 The LCS for batches of aqueous samples is prepared by adding 1.0 mL of the LCS spike solution to one liter of reagent water. The LCS for batches of aqueous samples is prepared by adding 0.025 mL of the LCS spike solution to one liter of reagent water for analyses. The LCS for batches of soil samples is prepared by adding 0.5 mL of the LCS spiking solution to 15 g of Sodium sulfate.

LCS Spiking Solution

Volume of AB Mix Stock (mL)	Conc. of AB Mix Stock (µg/mL)	Final Volume (mL)	Final Concentration (µg/mL)
0.100	2000	200	1.0

7.9 Matrix Spike (MS) Spike Solution, 1.0 µg/mL

7.9.1 The working matrix spike solution is the same as the LCS spike solution. Matrix spikes (MS and MSD) are prepared by adding 1.0 mL of the working spike solution to one liter of an aqueous sample, 0.025 mL of the working spike solution to one liter of an aqueous sample and 0.5 mL of the working spike solution to a 15-gram solid subsample.

7.10 Surrogate Spike Solution, 0.8 µg/mL for TCMX and 1.6 µg/mL for DCB

7.10.1 The surrogate stock solution, containing 200 µg/mL each of decachlorobiphenyl and tetrachloro-m-xylene (TCMX), is purchased from commercial sources.

7.10.2 The working surrogate spike solution is prepared in a volumetric flask by adding 0.8 mL of the TCMX stock solution and 1.6 mL of the DCB stock solution and diluting to a final volume of 200 mL with acetone.

7.10.3 For aqueous sample batches, 1.0 mL of the surrogate spike solution is added to each one-liter sample and QC sample. For low-level aqueous sample batches, 0.025 mL of the surrogate spike solution is added to each one-liter sample and QC sample. For soil sample batches, 0.5 mL of the surrogate spike solution is added to each 15-gram soil subsample and QC sample. For tissue sample batches, 0.2 mL of the surrogate spike solution is added to each 15-gram tissue subsample and QC sample.

7.11 Column Degradation Mix (EVAL B)

7.11.1 The DDT/endrin breakdown stock standard solution is obtained from commercial sources, with endrin at a concentration of 100 µg/mL, and 4,4'-DDT at 200 µg/mL.

7.11.2 The working EVAL B solution is prepared in a volumetric flask, by diluting 0.05 mL of the stock solution to 200 mL in hexane, as summarized in the following table:

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Column Degradation Mix (Eval B Std) Spike Solution			
Compound	**Volume of Stock (mL)**	**Final Volume (mL)**	**Final Concentration (µg/mL)**
Endrin	0.05	200	0.025
4,4'-DDT			0.05

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are collected in pre-cleaned, amber glass bottles fitted with a Teflon-lined cap. To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

8.2 Soil samples are collected in 8-ounce, pre-cleaned, wide-mouth jars with a Teflon-lined lid.

8.3 Samples are stored at 4 ± 2 °C.

8.4 Extracts are refrigerated at ≤ 6 °C.

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time
Waters	Amber glass	1 Liter	Cool 4 ± 2°C	7 Days	40 Days from extraction
Soils	Glass	30 grams	Cool 4 ± 2°C	14 Days	40 Days from extraction

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9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica PT-QA-021, Quality Control Program.

9.1.2 Refer to the TestAmerica Pittsburgh QC Program document (PT-QA-021) for further details on criteria and corrective actions.

9.1.3 For specific DoD quality control requirements refer to SOP # PT-QA-025, Implementation of the DoD QSM Version 3, January 2006 and Appendix I in this SOP. For DoD V4.1 refer to SOP PT-QA-029.

9.1.4 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.1.5 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP PT-QA-016. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank

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must be run on each instrument that is used to analyze samples from the same preparation batch. See QC SOP PT-QA-021 for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of 1.0 liter of reagent water, and for batches of soil samples, consists of 15 grams of sodium sulfate, both of which are free of any of the analyte(s) of interest. The method blank is processed and analyzed just as if it were a field sample.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above the reporting limit or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher. Wherever blank contamination is greater than 1/10 the concentrations found in the samples and/or 1/10 of the regulatory limit it is potentially at a level of concern and should be handled as a non-conformance. Blank contamination should always be assessed against project specific requirements. Such action must be taken in consultation with the client.

Corrective Action: Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.5 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which all the analyte(s) of interest are added at a known concentration. For soil sample batches, the LCS consists of sodium sulfate to which all the analyte(s) of interest are added at a known concentration. See Section 7.8 for the preparation of LCSs. The LCS is carried through the entire analytical procedure just as if it were a sample. TestAmerica Pittsburgh uses a full-analyte spike for its LCS. For specific DoD quality control requirements refer to SOP # PT-QA-025 and DoD QSM Appendix I in this SOP. For DoD V4.1 refer to SOP PT-QA-029.

See Table 7 for Laboratory generated Control Limits.

Acceptance Criteria: All analytes are spiked and list of control analytes are used to control the batch. The control analytes must meet the LCS control limits. For DoD follow SOP PT-QA-029. The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the

historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS. Corrective action will normally be repreparation and reanalysis of the batch.

Marginal Exeedance limits are not used unless required by a project. When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken. When using list of control compounds, all those compounds must be within acceptable limits.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. The recoveries for control analytes must be within the limit. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.5.1 Use of marginal exceedances is not permitted for South Carolina work for LCS. For SC work the LCS limits must be 70-130% at a maximum. Same limits or lab calculated limits

can be used for MS/MSD. If limits are calculated for LCS, they must be within 70-130 % range.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of all target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Refer to Section 7.9 for preparation of matrix spikes. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis. TestAmerica Pittsburgh uses a full-analyte spike for its MS/MSD. For specific DoD quality control requirements refer to SOP # PT-QA-025, Implementation of the DoD QSM Version 3, January 2006 and Appendix I in this SOP. For DoD V4.1 refer to SOP PT-QA-029.

See Table 7 for Laboratory generated Control Limits.

Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference (RPD) between the MS and MSD must be less than the established RPD limit, which is set at 3 standard deviations above the historical mean. Current control limits are maintained in the LIMS. For SC work lab generated limits can be used for MS/MSD or same limits as LCS can be used for MS/MSD, 70-130%.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e., method blank, LCS, LCSD, MS, and MSD) is spiked with DCB and TCMX surrogate compounds. Refer to Section 7.10 for preparation of the surrogate spike solution. See Table 7 for Laboratory generated Control Limits.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

For field samples and QC, both DCB and TCMX are evaluated. If one surrogate fails to fall within the control limits and the other surrogate is within the control limits, the data is considered reportable with an NCM and narration in the final report.

If a surrogate recovery fails, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Calibration and Standardization

10.1 TestAmerica Pittsburgh gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Use the ChemStation chromatography data system to set up GC conditions for calibration. See Table 2 for typical operating conditions.

10.1.2 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

10.1.3 Unprocessed calibration data are transferred to the TARGET DB database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the

Public Drive.

10.2 Column Degradation Evaluation

10.2.1 Each day of operation and at the beginning of each 12 hour shift, before any calibration or calibration verification standards are analyzed, the column degradation evaluation mix (EVAL B) must be analyzed. In addition, some programs require injection of the degradation evaluation mix more frequently. Unless otherwise specified in an approved project plan, the degradation check must be performed whether or not 4,4'-DDT, Endrin, or degradation compounds are designated as target analytes. The purpose of the evaluation is to determine whether instrument/column maintenance is needed. The preparation of this standard is described in Section 7.11.

10.2.2 The results of the analysis of the EVAL B standard solution are used to calculate column degradation in terms of DDT percent breakdown (%B) and Endrin %B as follows:

$$\text{DDT } \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100\% \qquad \textit{Equation 1}$$

Where A_{DDD}, A_{DDE}, and A_{DDT} are the peak responses for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT, respectively, in the EVAL B chromatogram.

$$\text{Endrin } \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100\% \qquad \textit{Equation 2}$$

Where A_{EK}, A_{EA}, and A_{E} are the peak responses for endrin ketone, endrin aldehyde, and endrin, respectively, in the EVAL B chromatogram.

10.2.3 Acceptance Criteria

10.2.3.1 The %D for each of these two compounds, 4,4'-DDT and Endrin, must be less than 15%

10.2.4 Corrective Action

10.2.4.1 If the breakdown of 4,4'-DDT and/or Endrin exceeds the 15% limit, corrective action must be taken. This action may include any or all of the following:

- Replacing the injection port liner or the glass wool.
- Cutting off a portion of the injection end of the column or guard column.
- Replacing the GC column or guard column
- Replacing the y-splitter.

10.2.4.2 After taking the appropriate corrective action, the degradation evaluation standard must be reanalyzed and must pass acceptance criteria before conducting any calibration events

10.3 The laboratory uses six calibration levels (as shown in Table 3) for the single-component pesticides. The lowest point on the calibration curve is at or below the reporting limit (RL). The highest standard defines the highest sample extract concentration that may be reported without dilution. The preparation of the calibration standards is described in Section 7.4.

10.4 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

Calibration for the multi-peak component analytes, Toxaphene and Technical Chlordane, is a single-point calibration. If requested by a client, a calibration for the multi-component analyte(s) can be conducted using a minimum of five calibration levels. The samples would then be analyzed using the full calibration curve that brackets the quantitation range. **For specific DoD quality control requirements refer to SOP # PT-QA-025, Implementation of the DoD QSM Version 3, January 2006 and Appendix I in this SOP.** For DoD V4.1 refer to SOP PT-QA-029.

NOTE: Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

10.4.1 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

- The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
- The lowest remaining calibration point is still at or below the project reporting limit; and
- The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
- The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average calibration factors or linear regressions, or six levels for second-order curve fits.

NOTE: Second order curves are not allowed for South Carolina work.

10.5 External Standard Calibration

10.5.1 External standard calibration involves the comparison of instrument responses (e.g., peak area or peak height) from the target compounds in the sample to the responses of the target compounds in the calibration standards. The ratio of the detector response to the amount or concentration of target analyte in the calibration standard is defined as the calibration factor (CF), as follows:

$$CF = \frac{A_S}{C_S} \qquad \textit{Equation 3}$$

Where:

A_s = Peak area (or height) of the analyte or surrogate in the calibration standard.

C_s = Concentration of the analyte or surrogate, in ng/mL, in the injected calibration standard.

10.6 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as calibration curves, using a systematic approach to selecting the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until the calibration acceptance criteria are met.

10.6.1 Linear Calibration Using Average Calibration Factor

Tabulate the peak area response for each target analyte in each calibration level against the concentration injected. For each analyte in each calibration standard, calculate the calibration factor (CF) as shown in Equation 3 above. The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

For each target analyte, calculate the average calibration factor as follows:

$$AverageCalibrationFactor = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \textit{Equation 4}$$

Where:

CF_i = Calibration factor for the ith calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\% \qquad \textit{Equation 5}$$

Where SD is the standard deviation of the average CF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \textit{Equation 6}$$

10.6.2 Evaluation of the Average Calibration Factor

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Note: The use of grand average (evaluation of the average response over all the compounds) is no longer allowed. Each compound must meet the RSD criteria.

Acceptance Criteria: The RSD must be ≤ 20%.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.6.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). A weighted least squares regression may be used if at least three multi-point calibrations have been performed. The weighting used is the reciprocal of the square of the standard deviation. The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b \qquad \textit{Equation 7}$$

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b \qquad \textit{Equation 8}$$

To calculate the concentration in an unknown sample extract, the regression equation (Equation 6) is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract.

$$C_e = \frac{A_e - b}{a} \qquad \textit{Equation 9}$$

Where:

A_s = Area of the chromatographic peak for the target analyte in the calibration standard.

A_e = Area of the chromatographic peak for the target analyte in the sample extract.

a = Slope of the line as determined by the least-squares regression.

C_s = Concentration of the target analyte in the calibration standard.

C_e = Concentration of the target analyte in the sample extract.

b = Intercept of the line as determined by the least-squares regression.

10.6.4 Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum “Clarification Regarding Use of SW-846 Methods”, Attachment 2, Page 9, includes the statement “The Agency further recommends the use of a weighted regression over the use of an unweighted regression.”

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit, and preferably less than the MDL.

Also examine the residuals, paying particular attention to the residuals at the low end of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

The linear regression must have a correlation coefficient (r) ≥ 0.990. Some programs (e.g., DoD) require a correlation coefficient ≥ 0.995.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

10.6.5 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c \qquad \textit{Equation 10}$$

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

10.6.6 Non-Linear Calibration Evaluation

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria: The coefficient of determination must be ≥ 0.990.

Second-order regressions should be the last option. **Note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.**

Before selecting a second-order regression calibration model, it is important to ensure the following:

- The absolute value of the intercept is not large relative to the lowest concentrations being reported.
- The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).
- The distribution of concentrations is adequate to characterize the curvature.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem must be investigated and the instrument recalibrated. **Third-order regressions are not allowed at TestAmerica Pittsburgh.**

10.7 Initial Calibration Verification (ICV), 0.025 µg/mL for most compounds

A mid-level standard that is obtained from a source different from that of the calibration standards (second-source standard) is used to verify the initial calibration. The ICV standard is analyzed immediately following the initial calibration (ICAL).

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 20% of the expected value(s).

Corrective Action: If this is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.8 Calibration Verification

10.8.1 12-Hour Calibration Verification

10.8.1.1 The 12-hour calibration verification sequence consists of, at a minimum, an instrument blank and the mid-level calibration standard. The 12-hour calibration verification sequence must be analyzed within 12 hours of the initial calibration and at least once every 12 hours thereafter when samples are being analyzed.

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.8.2 Continuing Calibration Verification (CCV), 0.020 µg/mL for most compounds

10.8.2.1 It may be appropriate to analyze a mid-level standard, Levels 3, 4 or 5, more frequently than every 12 hours. The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard (see Section 7.6). At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCSs, and method blanks. A CCV is also analyzed at the end of each analytical sequence. Some programs require a CCV after every 10 samples to minimize the number of samples requiring re-injection when QC limits are exceeded. If 12 hours elapse, analyze the 12-hour standard sequence instead.

10.8.3 RL Standard

10.8.3.1 It may also be appropriate to analyze a standard prepared at or very near the

reporting limit (RL) for the method at the end of the analytical sequence, as a minimum (see Section 7.7). This standard can be used to rule out false negatives in client samples in cases where the %D for one or more of the analytes in a bracketing CCV falls below the lower acceptance limit. The results for the RL standard are not evaluated unless the previous CCV fails acceptance criteria.

10.8.4 Acceptance Criteria for Continuing Calibration Verification (CCV)

10.8.4.1 Detected Analytes (≥ RL)

For any analyte detected at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration, on the column used for quantitation, must be within ± 20%.

In some cases, the nature of the samples being analyzed may be the cause of a failing %D. When the %D for an analyte falls outside of ± 20% in the CCV, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect if so desired by the client.

10.8.4.2 Non-Detected Analytes (≤ RL)

However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., >20%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the verification standard has demonstrated that the analyte would have been detected were it present. If an analyte was not detected in the sample and the standard response is more than 20% below the initial calibration response, then reinjection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present (i.e., a false negative result).

Refer to Section 12 for which result to report.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100$$ *Equation 11*

10.9 Retention Time Windows

10.9.1 Retention time (RT) windows must be determined for all analytes.

10.9.2 Make an injection of all analytes of interest each day over a 72-hour period.

10.9.3 Calculate the mean and standard deviation for the three RTs for each analyte as follows:

$$\text{Mean RT} = \overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RT_i - \overline{RT}\right)^2}{n-1}}$$

Equations 12 & 13

Where:

RT_i = Retention time for the ith injection.

n = Number of injections (typically 3).

SD = Standard deviation.

NOTE: For the multi-component analytes, Toxaphene and Technical Chlordane, the mean and standard deviation must be calculated for each of the 3 to 6 major peaks used for sample calculations.

10.9.4 Set the width of the RT window for each analyte at ± 3 standard deviations of the mean RT for that analyte.

10.9.5 The center of the RT window for an analyte is the RT for that analyte from the last of the three standards measured for the 72-hour study.

10.9.6 The center of the window for each analyte is updated with the RT from the Level 3 standard of the ICAL, or the CCV at the beginning of the analytical sequence. The width of each window remains the same until new windows are generated following the installation of a new column, or in response to an RT failure. The RT window width may be expanded if the RT drift observed in the ICAL is greater than the established window. The expanded window is noted on the ICAL checklist

10.10.6 If the RT window as calculated above is less than ± 0.01 minute, use ± 0.01 minute as the RT window. This allows for slight variations in retention times caused by sample matrix. Typically ± 0.05 minutes is used as the RT window.

10.10.7 TestAmerica Pittsburgh typically updates the retention time windows based on the previous CCV and would only determine a new RT window if a different phase column than those mentioned in Section 6 is used. The laboratory monitors the retention time for both surrogates throughout the analytical sequence.

11 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP PT-QA-016. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Preparation

11.3.1 Sample preparation for aqueous, soil, sediment, tissue, waste and oil samples is described in SOP PT-OP-001.

11.3.2 Cleanup and concentration of sample extracts is described in SOP PT-OP-001.

11.3.3 The final extract volume in hexane is dependent on matrix (see Table 8).

11.3.4 Use hexane to dilute sample extracts, if necessary.

11.4 Gas Chromatography

Chromatographic conditions for this method are presented in Table 2. Use the ChemStation interface to establish instrument operating conditions for the GC. Raw data obtained by the ChemStation software is transferred to the TARGET DB database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the TARGET DB software.

11.5 Sample Introduction

All extracts and standards are allowed to warm to room temperature before injection. An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract. Samples, standards, and QC samples must be introduced using the same procedure. Use the ChemStation interface to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

11.6 Analytical Sequence

An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. Refer to Table 3 for the calibration levels used.

11.6.1 Prior to analyzing any calibration or calibration verification standards, the column

degradation evaluation standard is injected and the results are evaluated as described in Section 10.2.

11.6.2 The daily calibration verification includes analysis of the 12-hour calibration sequence (Section 10.8.1) and updating the retention time windows (see Section 10.9).

11.6.3 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration verification.

11.6.4 The following is a typical analytical sequence:

- Solvent blank (optional)
- Eval B Std (column degradation evaluation)
- Toxaphene (Level 3[1])
- Technical Chlordane (Level 3[1])
- Pesticide Mix (All Levels)
- ICV (2nd source - all single component analytes)
- Eval B Std (column degradation evaluation)
- Solvent Blank (optional)
- Up to 20 samples (unless 12 hours comes first)[2]
- Pesticide Mix (CCV's - Level 3, 4 or 5[1])
- Eval B
- Up to 20 samples (unless 12 hours comes first)[2]
- Pesticide Mix (CCV's - Level 3, 4 or 5[1])
- Eval B

[1]A five-point curve for any of the multicomponent analytes may be included, if requested by the client.

[2]At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Pesticide Mix, and the breakdown mix must be run before the continuing calibration.

11.7 Daily Retention Time Windows

The center of the retention time (RT) windows determined in Section 10.9 are adjusted to the RT of each analyte as determined in the 12-hour calibration verification. The centers of the RT windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration, but not for any other calibration verification standards.

11.8 Upon completion of the analytical sequence, transfer the raw chromatography data to the TARGET DB database for further processing.

11.8.1 Review chromatograms online and determine whether manual data manipulations are necessary.

11.8.2 All manual integrations must be justified and documented. See CA-Q-S-002 for requirements for manual integration.

11.8.3 Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

11.8.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

11.8.5 Case Narrative: For DoD the case narrative shall provide: identification of **samples and analytes** for which manual integration was necessary. DoD QSM, Version 3, Appendix DoD-A. For DoD V4.1 refer to SOP PT-QA-029.

11.9 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.9.1 Perform a level 1 data review and document the review on the data review checklist (GC and HPLC Data Review Checklist).

11.9.2 Submit the data package and review checklist to the peer reviewer for the Level 2 review process. The data review process is explained in SOP PT-QA-018.

12 Calculations / Data Reduction

12.1 Qualitative Identification

12.1.1 Tentative identification of an analyte occurs when a peak is found on the primary column within the RT window for that analyte, at a concentration above the reporting limit, or above the MDL if qualified data (J flags) are to be reported. Identification is confirmed if a peak is also present in the RT window for that analyte on the second (confirmatory) column and if the analyte concentration is greater than the MDL. When confirmation is

made using a second column, the analysis on the second column must meet all of the QC criteria for continuing calibration verification and RTs.

12.1.2 The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2 Dual-Column Quantitation and Reporting

12.2.1 Each sample is analyzed on two different columns at the same time. The laboratory designates a primary column based on optimal separation of the compounds of interest and other desirable chromatographic characteristics. The higher of the two results is normally if the percent difference between the responses of the two columns or detectors is within 40%. **For DoD QSMV3.0, report the higher of the two confirmed results unless overlapping peaks are causing erroneously high results, then report the nonaffected result and document in the case narrative**. For DoD V4.1 refer to SOP PT-QA-029. The result from the secondary (confirmatory) column is reported if any of the following is true:

- There is obvious chromatographic interference on the primary column.
- The difference between the result for the primary column and the result for the secondary column is > 40% and chromatographic interference is evident.
- The continuing calibration verification, bracketing standard, or surrogate recovery fails on the primary column, but is acceptable on the secondary column. However, if the difference between the primary column result and the secondary column result is > 40% and the primary column calibration fails, then the sample must be evaluated for reanalysis.

12.2.2 Dual Column Results With > 40% RPD

12.2.2.1 If the Percent Difference (%D) between the response on the two columns or detectors is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. If the CCV % D is within ± 20% for one column and >20% for the other column and the %D between the two columns is within 40%, the data is reported from the column with the CCV within ± 20%.

12.2.2.2 The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\% \qquad \textit{Equation 14}$$

Where R_1 is the result for the first column and R_2 is the result for the second column.

12.3 Multi-Component Analytes (Toxaphene and Technical Chlordane)

12.3.1 Qualitative Identification

Retention time windows are also used for identification of multi-component analytes, but the “fingerprint” produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3.2 Quantitation of Toxaphene

12.3.2.1 While Toxaphene contains a large number of compounds that produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. The unresolved complex mixture results in a "hump" in the chromatogram that is characteristic of the Toxaphene mixture of compounds. The resolved peaks are important for the identification of the mixture, and the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

12.3.2.2 To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the RTs of the first and last eluting Toxaphene components in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated.

12.3.2.3 Toxaphene may also be quantitated on the basis of 4 to 6 major peaks. Using a subset of 4 to 6 peaks for quantitation provides results that agree well with the total peak approach and may avoid difficulties when interferences with Toxaphene peaks are present in the early portion of the chromatogram from compounds such as DDT.

12.3.2.4 When Toxaphene is determined using the 4 to 6 peaks approach, care must be taken to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms.

12.3.2.5 The chosen peaks must be within the established retention time. If there is an interference that affects the accuracy of results, the analyst may use as few as 4 major peaks. The same peaks that are used for sample quantitation must be used for calibration.

12.3.2.6 The heights or areas of the chosen peaks should be summed together to determine the Toxaphene concentration.

12.3.2.7 Second column confirmation of multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: USACE projects require the use of second-column confirmation of multi-component analytes unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

12.3.3 Quantitation of Technical Chlordane

12.3.3.1 Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. Cis-Chlordane (or α-Chlordane) and trans-Chlordane (or γ-Chlordane) are the two most prevalent major components of Technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch.

12.3.3.2 When the GC pattern of the sample resembles that of Technical Chlordane, Chlordane may be quantitated by comparing the total area of the Chlordane chromatogram using 3 to 5 major peaks or the total area. If the Heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation. If Heptachlor and/or Heptachlor epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected Chlordane area.

NOTE: Octachlor epoxide, a metabolite of Chlordane, can easily be mistaken for Heptachlor epoxide on a nonpolar GC column.

12.3.3.3 To measure the total area of the Chlordane chromatogram, construct the baseline of Technical Chlordane in each calibration chromatogram between the RTs of the first and last eluting Technical Chlordane components. Use this area and the mass or concentration of Technical Chlordane in each calibration standard to establish the calibration function (Section 10.6). Construct a similar baseline in the sample chromatogram, measure the area, and use the calibration function to calculate the concentration in the sample extract.

12.3.3.4 When the GC pattern of Chlordane in a sample differs considerably from that of the Technical Chlordane standard, it may not be practical to relate a sample chromatogram back to the Technical Chlordane standard chromatogram. In these cases, all identifiable Chlordane components may be summed and reported as "Chlordane (not otherwise specified, CAS number 57-74-9)."

12.3.3.5 A third option for quantitating Technical Chlordane is to quantitate the peaks for α-Chlordane, γ-Chlordane, and Heptachlor separately against the appropriate reference materials, and report these individual components under their respective CAS numbers.

12.3.4 Second column confirmation of multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence. **NOTE: All South Carolina samples will be analyzed on a confirmation column.**

NOTE: USACE projects require the use of second-column confirmation of multi-component analytes unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

12.4 Surrogate recovery results are calculated and reported for tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in all samples and QC. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.5 Calibration Range and Sample Dilutions

12.5.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for the analyte(s) that were found to be over the calibration range in the high sample, they must be reanalyzed to rule out carryover. It may also be necessary to dilute samples because of matrix interferences.

12.5.2 If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.5.3 Guidance for Dilutions Due to Matrix Interference
If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard. Dilutions that are between 3X and 5X, report the data and narrate the dilution. Dilutions that are greater than 5X are reported as diluted out.

12.5.4 Reporting Dilutions

Some programs (e.g., South Carolina) and some projects require reporting of multiple dilutions (check special requirements in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported.

12.6 Interferences Observed in Samples

12.6.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants. If peak detection is prevented by interferences, further cleanup should be attempted (see SOP PT-OP-001). If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.7 Calculations

12.7.1 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\% \qquad \textit{Equation 15}$$

12.7.2 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD } \%\text{Recovery} = \left(\frac{SSR - SR}{SA}\right) \times 100\% \qquad \textit{Equation 16}$$

Where:

SSR = Measured concentration in spiked sample.

SR = Measured concentration in unspiked sample.

SA = Concentration of spike added to sample.

12.7.3 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\% \qquad \textit{Equation 17}$$

Where *R1* is the result for the MS and *R2* is the result for the MSD.

12.7.4 Concentration of Analyte in the Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.6 for details on establishing the calibration function):

Average Calibration Factor: $$C_e = \frac{A_s}{\overline{CF}} \qquad \textit{Equation 18}$$

Linear Regression: $$C_e = \frac{[A_s - b]}{a} \qquad \textit{Equation 19}$$

Non-Linear Regression: $$C_e = f(A_s) \qquad \textit{Equation 20}$$

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_s = Peak area for the analyte in the sample extract injection.

B = y-intercept of the calibration fit.

A = Slope of the calibration fit.

$f(A_s)$ = Mathematical function established by the non-linear regression.

12.7.5 Concentration of Analyte in Original Sample

$$C_{sample} = \frac{C_e}{1000\frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF \qquad \textit{Equation 21}$$

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e = Concentration of analyte in sample extract injected in GC (ng/mL).

$1000\frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.

V_e = Volume of sample extract (mL).

V_s = Volume (or weight) of original sample (L or kg).

DF = Dilution Factor (post extraction dilutions)

12.8 All data are subject to two levels of review, which is documented on a checklist, as described in SOP DEN-QA-0020.

13.0 Method Performance

13.1 Initial Demonstration of Capability

An initial demonstration of capability for each method must be performed prior to analyzing samples.

13.1.1 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample (e.g., LCS) containing all of the standard analytes for the

method, as well as a method detection limit (MDL) study (described in Section 13.2 below).

13.1.2 Four aliquots of the LCS sample are analyzed with the same procedures used to analyze samples, including sample preparation.

13.1.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria (e.g., LCS control limits). All four results must meet acceptance criteria before the method can be used to analyze samples.

13.1.4 For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

13.2 Method Detection Limit (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy PT-QA-007. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

13.3 Analyst Training and Qualification

13.3.1 The Group/Team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP PT-QA-021.

13.3.2 Each analyst performing the method must complete an initial demonstration of capability (IDOC) by successfully preparing and/or analyzing four consecutive LCSs or other acceptable QC samples. The results of the IDOC study are summarized in the NELAC format, as described in SOP PT-QA-021. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

14.0 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Safety Manual, and PT-HS-001, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Methylene Chloride in vials. This waste is placed in waste container identified as "Vials & Extracts", Waste #7.

15.2.2 Flammable solvents in vials. This waste is placed in waste container identified as "Vials & Extracts", Waste #7.

15.2.3 Waste flammable solvents. This waste is collected in a waste container identified as "Mixed Flammable Solvent Waste", Waste #3.

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16 References / Cross- References

16.1 Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatograph, Revision 1, February, 2007, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December, 1996, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.3 SOP PT-OP-001, Extraction and Cleanup of Organic Compounds from Waters and Solids, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series Methods, current version.

16.4 SOP PT-QA-001, Employee Orientation and Training, current version.

16.5 SOP CA-Q-S-002, Manual Integration, current version.

16.6 SOP PT-QA-007, Determination of Method Detection Limits (MDLs), current version.

16.7 SOP PT-QA-016, Nonconformance & Corrective Action System, current version.

16.8 SOP PT-QA-018, Technical Data Review Requirements, current version.

16.9 SOP PT-QA-021, Quality Control Program, current version.

16.10 SOP PT-QA-025, Implementation of the DoD QSM Version 3, January 2006, current version.

16.11 SOP PT-QA-029, DoD Version 4.1 Requirements, current version.

16.12 PT-LQAM, Pittsburgh Laboratory Quality Assurance Manual, current version.

17 Method Modifications:

Item	Method	Modification
1	8081B	Method 8081B includes an internal standardization option. Because of the high probability of interferences affecting internal standards, this SOP allows only external standards.
2	8081B	Section 11.4.1.1, allows the use of a single-point calibration for the multi-component pesticides. In this SOP an initial single-point calibration is used, but a five-point calibration followed by reanalysis of associated samples is required when one of the multi-component pesticides is detected.
3	8081B	Method 8081B references 8000B, which allows the use of third-order calibration curves. TestAmerica Pittsburgh does not allow third-order curves.

18 Tables

Table 1: Analyte List and Standard Reporting Limits

Table 2: Typical Instrument Conditions

Table 3: Calibration Levels ((µg/mL)

Table 4: Column Degradation Evaluation Mix

Table 5: LCS/Matrix Spike and Surrogate Spike Levels

Table 6: Evaluation Criteria and Corrective Actions for Continuing Calibration Verification

Table 7: LCS and MS/MSD Control Limits

Table 8: Initial Volumes/Weights/Exchange Solvents and Final Volumes for Pesticides

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19 Revision History

19.1 Revision 1.

19.1.3 Added Section 9.5.1: Use of marginal exceedances is not permitted for South Carolina work for LCS. For SC work the LCS limits must be 70-130% at a maximum. Same limits or lab calculated limits can be used for MS/MSD. If limits are calculated for LCS, they must be within 70-130% range. Section 9.6 for updated for SC requirements. LCS and MS control limits corrected in Table 7.

19.2 Revision 2:

19.2.3 Updated section 9.5 to include use of control analytes. ME limit will only be used if required by a project. For DoD QSM 4.1 follow SOP PT-QA-029.

19.3 Revision 3

19.3.3 Section 9.5 was corrected to show that sodium sulfate not sodium thiosulfate is used for the LCS for soil analysis.

19.3.4 In Section 10.8.2.1 a statement was added to indicate that a CCV is analyzed at the end of each analytical sequence.

19.3.5 In Section 12.3.4 a statement was added to indicate that South Carolina samples will be analyzed on a confirmation column.

Table 1.
Analyte List and Standard Reporting Limits
µg/L, µg/wipe or µg/kg

Compound	CAS#	Water or Wipe/TCLP	Soil/ Low Level Soil	Tissue	Waste	Low Level Water
Aldrin	309-00-2	0.05	1.7/0.0833	1.7	50	0.0013
α-BHC	319-84-6	0.05	1.7/0.0833	1.7	50	0.0013
β-BHC	319-85-7	0.05	1.7/0.0833	1.7	50	0.0013
δ-BHC	319-86-8	0.05	1.7/0.0833	1.7	50	0.0013
γ-BHC (Lindane)	58-89-9	0.05/0.5	1.7/0.0833	1.7	50	0.0013
α-Chlordane	5103-71-9	0.05	1.7/0.0833	1.7	50	0.0013
γ-Chlordane	5103-74-2	0.05	1.7/0.0833	1.7	50	0.0013
Chlordane (tech.)	57-74-9	0.5/5.0	17/0.0833	17	500	0.0125
4,4'-DDD	72-54-8	0.05	1.7/0.0833	1.7	50	0.0013
4,4'-DDE	72-55-9	0.05	1.7/0.0833	1.7	50	0.0013
4,4'-DDT	50-29-3	0.05	1.7/0.0833	1.7	50	0.0013
Dieldrin	60-57-1	0.05	1.7/0.0833	1.7	50	0.0013
Endosulfan I	959-98-8	0.05	1.7/0.0833	1.7	50	0.0013
Endosulfan II	33213-65-9	0.05	1.7/0.0833	1.7	50	0.0013
Endosulfan Sulfate	1031-07-8	0.05	1.7/0.0833	1.7	50	0.0013
Endrin	72-20-8	0.05/0.5	1.7/0.0833	1.7	50	0.0013
Endrin Aldehyde	7421-93-4	0.05	1.7/0.0833	1.7	50	0.0013
Endrin ketone	53494-70-5	0.05	1.7/0.0833	1.7	50	0.0013
Heptachlor	76-44-8	0.05/0.5	1.7/0.0833	1.7	50	0.0013
Heptachlor Epoxide	1024-57-3	0.05/0.5	1.7/0.0833	1.7	50	0.0013
Methoxychlor	72-43-5	0.1/1.0	3.3/0.1666	3.3	100	0.0025
Toxaphene	8001-35-2	2.0/20	67/3.333	67	2000	0.0500
NON ROUTINE STANDARDS*						
2,4'-DDE	3424-82-6	0.05	1.7	--	50	0.0013
2,4'-DDD	53-19-0	0.05	1.7	--	50	0.0013
Cis-Nonachlor	5103-73-1	--	1.7	--	--	0.0013
Trans-Nonachlor	39765-80-5	--	1.7	--	--	0.0013
Hexachlorobutadiene	87-68-3	--	1.7	--	--	--
2,4'-DDT	789—02-6	0.05	1.7	--	50	0.0030
Chlorbenside	103-17-3	0.1	3.3	3.3	100	0.0032
Dacthal (DCPA)	1861-32-1	0.1	3.3	3.3	100	0.0025
Hexachlorobenzene	118-74-1	0.05	1.7	--	50	0.0013
Hexachlorocyclopentadiene	77-47-4	0.1	3.3	--	100	--
Mirex	2385-85-5	0.05	1.7	1.7	50	0.0013
Diallate	2303-16-4	1.0	33	--	990	0.025

Table 1. Analyte List and Standard Reporting Limits µg/L, µg/wipe or µg/kg						
Compound	**CAS#**	**Water or Wipe/TCLP**	**Soil/ Low Level Soil**	**Tissue**	**Waste**	**Low Level Water**
Isodrin	465-73-6	0.05	1.7	--	50	0.0013
Chlorobenzillate	510-15-6	0.5	17	--	500	0.0373

* These are non-routine compounds that require a separate calibration, and are analyzed only upon request.

The following concentration factors are used when calculating the Reporting Limits:

	Extraction Vol.	**Final Vol.**
Groundwater	1000 mL	40 mL (1 mL for low-level)
TCLP Leachate	100 mL	40 mL
Soil	15 g	20 mL (1 mL for low-level)
Wipe	1 wipe	40 mL
High-Level Solid Waste	1 g	40 mL
Tissue	5 g	1 mL (with GPC clean-up)

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Table 2. Typical Instrument Conditions	
Parameter	**Recommended Conditions***
Injection port temperature	220 ^{o}C
Detector temperature	325 ^{o}C
Column 1 (HP6890 GC)	MR1: 30 m X 0.53 mm id, 0.5 µm
Column 2 (HP6890 GC)	MR2: 30 m X 0.53 mm id, 0.5 µm
Temperature program	120 ^{o}C for 1 minute 8.5 ^{o}C/min to 285 ^{o}C and hold for 6 minute
Injection	2 µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

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Table 3.
Calibration Levels (ng/mL)

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix A and B						
Aldrin	1	5	25	50	100	200
g-BHC (Lindane)	1	5	25	50	100	200
Heptachlor	1	5	25	50	100	200
Methoxychlor	2	10	50	100	200	400
Dieldrin	1	5	25	50	100	200
Endosulfan I	1	5	25	50	100	200
Endosulfan II	1	5	25	50	100	200
4,4'-DDT	1	5	25	50	100	200
Endrin Aldehyde	1	5	25	50	100	200
Endrin ketone	1	5	25	50	100	200
β-BHC	1	5	25	50	100	200
δ-BHC	1	5	25	50	100	200
α-BHC	1	5	25	50	100	200
4,4'-DDD	1	5	25	50	100	200
4,4'-DDE	1	5	25	50	100	200
Endosulfan Sulfate	1	5	25	50	100	200
Endrin	1	5	25	50	100	200
α-Chlordane [3]	1	5	25	50	100	200
γ-Chlordane [3]	1	5	25	50	100	200
Heptachlor Epoxide	1	5	25	50	100	200
Appendix IX Standards						
Chlorobenside	2	10	250	100	200	400
Dacthal	2	10	250	100	200	400
Hexachlorobenzene	1	5	125	50	100	200
Hexachlorocyclopentadiene	2	10	250	100	200	400
Isodrin	0.4	2	50	20	40	20
Mirex	0.8	4	100	40	80	40
Oxychlordane isomer	0.4	2	50	20	40	20
Multicomponent Standards						
Chlordane (Technical)			250[4]			
Toxaphene			1000[5]			
Surrogates are included the AB Mix calibration mix at the following levels:						
Tetrachloro-m-xylene	1	5	25	50	100	200

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Table 3. Calibration Levels (ng/mL)						
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6[2]**
Decachlorobiphenyl	1	5	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.

[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

[4] This standard may be used for quantitation of technical chlordane between 50 and 1000 ng/mL. If the chlordane is more concentrated, the extract must be diluted and reanalyzed.

[5] This standard may be used for quantitation of toxaphene between 200 and 4000 ng/mL. If the toxaphene is more concentrated, the extract must be diluted and reanalyzed

Table 4. Column Degradation Evaluation Mix	
Component	**Concentration (ng/mL)**
4,4'-DDT	50
Endrin	25
TCMX (surrogate)	20
DCB (surrogate)	20

Table 5. LCS/Matrix Spike and Surrogate Spike Levels µg/L, µg/wipe or µg/kg		
Compound	**Aqueous/Wipe**	**Soil/Tissue**
Aldrin	0.25	8.33
g-BHC (Lindane)	0.25	8.33
Heptachlor	0.25	8.33
Methoxychlor	0.25	8.33
Dieldrin	0.25	8.33
Endosulfan I	0.25	8.33
Endosulfan II	0.25	8.33
4,4'-DDT	0.25	8.33
Endrin Aldehyde	0.25	8.33
Endrin ketone	0.25	8.33
β-BHC	0.25	8.33
δ-BHC	0.25	8.33
α-BHC	0.25	8.33
4,4'-DDD	0.25	8.33
4,4'-DDE	0.25	8.33
Endosulfan Sulfate	0.25	8.33
Endrin	0.25	8.33
α-Chlordane [3]	0.25	8.33
γ-Chlordane [3]	0.25	8.33
Heptachlor Epoxide	0.25	8.33
Surrogates		
Decachlorobiphenyl	0.2	6.67
Tetrachlor-m-xylene (TCMX)	0.2	6.67

Table 6. LCS/Matrix Spike and Surrogate Spike Levels for TCLP (µg/L)	
Heptachlor	2.5
Heptachlor epoxide	2.5
g-BHC (Lindane)	2.5
Endrin	2.5
Methoxychlor	2.5
TCMX (surrogate)	2.0
DCB (surrogate)	2.0

Table 7. LCS and MS/MSD Control Limits[1]						
	Water			Solid		
	LCS and MS/MSD			LCS and MS/MSD		
Compound	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Aldrin ©	69	121	22	70	123	22
α-BHC	55	133	20	59	127	20
β-BHC	71	129	24	70	128	20
δ-BHC	30	137	26	40	124	20
g-BHC (Lindane) ©	63	123	20	66	124	20
α-Chlordane	67	127	21	71	130	20
γ-Chlordane	65	121	21	68	123	24
Chlordane (technical)	47	143	41	33	150	40
4,4'-DDD	76	128	24	70	135	20
2,4'-DDD	50	150	20	50	150	20
4,4'-DDE	74	125	20	70	133	20
2,4'-DDE	50	150	20	50	150	20
4,4'-DDT ©	62	160	24	61	126	37

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Table 7.
LCS and MS/MSD Control Limits[1]

	Water			Solid		
	LCS and MS/MSD			LCS and MS/MSD		
2,4'-DDT	50	150	20	50	150	20
Dieldrin ©	70	123	20	70	123	20
Endosulfan I	72	123	22	70	126	23
Endosulfan II	72	122	21	70	128	33
Endosulfan Sulfate	58	129	21	55	129	26
Endrin ©	70	125	24	70	127	20
Endrin Aldehyde	61	116	25	65	122	20
Endrin ketone	73	123	20	70	132	20
Heptachlor ©	65	127	25	70	128	20
Heptachlor Epoxide	71	122	20	69	131	20
Hexachlorobenzene	50	150	20	50	150	20
Methoxychlor	59	143	27	70	143	26
Mirex	40	135	30	40	135	30
trans-Nonachlor	50	150	20	50	150	20
Toxaphene	30	150	30	30	150	30
Decachlorobiphenyl (surrogate)	45	130	--	45	130	--
TCMX (surrogate)	45	130	--	45	130	--

[1]The Control Limits are subject to change as in-house limits are evaluated and updated by the QAM.

© Denotes control compounds. For DoD see SOP PT-QA-029.

Table 8.
Initial Volumes/ Weights, Exchange Solvents and Final Volumes[1] for Pesticides

Matrix	Initial Volume/Weight	Exchange Solvent for Analysis	Final Volume for Analysis (mL)
Water	1000 mL	Hexane	40.0
Soil/Sediment	15 g	Hexane	20.0
Tissue	6 g	Hexane	8.0
Low Level Analyses	1000 mL/ 15 g	Hexane	1.0

[1] Final Volumes will be ½ of the volume specified under Final Volume for Analysis if GPC Cleanup is performed (¼ if both Soxtherm® and GPC performed). GPC is required for all tissue analyses except PCBs, where it is recommended but optional if acid cleanup is performed.

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POLYCHLORINATED BIPHENYLS (PCB) AS AROCLORS BY GAS CHROMATOGRAPHY (GC)

1 SCOPE AND APPLICATION

1.1 This method is used for the determination of PCBs as Aroclors in aqueous matrices, solid matrices, and wipes. The following compounds can be determined by this method:

Target Mixture	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

1.2 Instrumentation: HP 5890 Gas Chromatograph (GC) equipped with an electron capture detector (ECD) and a LEAP Technologies CTC A200S autosampler and Agilent Chemstation Data Acquisition software

1.3 Compound Target Mixtures are all part of a class of compounds known as Aroclors. The presence of an Aroclor will result in a specific GC-ECD chromatogram pattern with multiple peaks.

1.4 When the method is used to analyze unknown samples, Aroclor identification is done by retention time and pattern recognition. This method describes identification of specific Aroclors by pattern recognition.

1.5 Due to the complex nature of the multi-response Aroclor patterns, this method is restricted to the use by or under the supervision of analysts experienced in the use of a gas chromatograph and skilled in the interpretation of chromatograms.

2 SUMMARY OF METHOD

2.1 The method provides for the extraction and GC conditions for the analysis of PCB Aroclors in aqueous and solid matrices, and on wipes.

2.2 Soil samples are dried with sodium sulfate and extracted with a mixture of iso-octane/acetone. Water samples are saturated with sodium chloride and extracted with iso-octane or by using separatory funnel extraction with dichloromethane. Wipe samples are extracted with a mixture of iso-octane/acetone.

2.3 Extracts are transferred to injection vials and analyzed by GC-ECD.

2.4 The sensitivity and identification issues associated with this method depend on level of matrix interferences in addition to instrumental limitations. If interferences are

present, the resulting report limit may have to be elevated. Table 1 lists LODs that can be obtained in water, sand, and wipes in the absence of interferences.

2.5 A standard curve is generated for one of the Aroclors from a minimum six point standard curve using power regression as the regression analysis equation. The other Aroclor mixtures are quantitated using a single point calibration. Quantitation for each target mixture is done using reverse extrapolation.

3 DEFINITIONS AND ACRONYMS

3.1 There are many terms and acronyms used throughout this document. Check the definitions and acronyms sections of the Quality Manual for complete explanations.

4 INTERFERENCES

4.1 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts, column surfaces or detector surfaces; and the presence of co-eluting compounds in the sample. Interferences co-extracted from the samples will vary considerably from sample to sample. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

4.2 Interferences by phthalates introduced during sample preparation can pose a major problem in PCB determinations. Common flexible plastics contain varying amounts of phthalates, which are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interferences from phthalate esters. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate ester contamination.

4.3 Glassware must be scrupulously cleaned if other than disposable glassware is used. Clean all glassware as soon as possible after use by detergent washing with hot water, three rinses with hot tap water, and three rinses with organic-free reagent water. Immediately prior to use, all non-disposable glassware/apparatus is rinsed with dichloromethane or another appropriate solvent.

4.4 Colored sample extracts should be cleaned with sulfuric acid prior to instrument analysis in accordance with PRE-004, Sulfuric Acid Clean-up.

4.5 The presence of elemental sulfur will result in a broad peak that interferes with the detection of and quantitation of PCB's. Sulfur contamination should be expected with sediment samples. Mercury cleanup is suggested for removal of sulfur in accordance with PRE-005, Mercury Clean-up.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the ECCS Chemical Hygiene Plan (CHP), and this document. Refer to the CHP for more detailed safety information or for information not listed in this document.

5.2 Eye protection that protects against splash and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled during this procedure. Lab coats are recommended.

5.3 Employees must handle glassware and equipment carefully in order to prevent injury and accidents. Any damaged or broken glassware is to be discarded or moved to the glass repair box.

5.4 ECCS maintains a Material Safety Data Sheet (MSDS) for every chemical used in the laboratory. The MSDS file is kept in the main laboratory.

6 APPARATUS AND MATERIALS

6.1 Gas Chromatograph (GC)

6.1.1

Gas Chromatograph:	HP5890
Autosampler:	LEAP Technologies A200S
Detector:	HP Electron Capture
Injector:	Split/splitless Injector
Data system:	Agilent Chemstation chromatography data system using HPIB capable of presenting chromatograms, retention time, peak integration data and calculating standard curves using regression analysis.

6.1.2 Columns

6.1.2.1 Column 1: RTX-35, 30 m x 0.53 mm ID, 0.5 µm film thickness or equivalent

NOTE: Primary column: Most Aroclor quantitation and identification is done utilizing this column.

6.1.2.2 Column 2: RTX-1701, 30 m x 0.53 mm ID, 0.25 µm film thickness or equivalent

NOTE: Backup column in the rare case where further identification is required.

6.1.3 GC supplies

6.1.3.1 High temperature septa, Restek part #27106

6.1.3.2 Injection port liners: Gooseneck 4 mm ID deactivated, Restek part # 20800

6.1.3.3 Gold seals, Restek part # 20476

6.2 Balances:

6.2.1 Top loader balance capable of weighting to 0.01 g

6.2.2 Analytical balance capable of weighting to 0.0001 g

6.3 Vials:

6.3.1 2 mL amber injection vials with Teflon lined crimp seals

6.3.2 200 µl inserts for 2 mL injection vials

6.3.3 20 mL scintillation vials

6.4 Syringes: Gas-tight, various sizes

6.5 Disposable glass transfer pipettes and 2 mL rubber bulbs

6.6 Glass funnel, 60-90 mm

6.7 Graduated glass cylinders, various sizes

6.8 Compressed Gas

6.8.1 Helium, Grade 5

6.8.2 Nitrogen, Grade 5

6.8.3 Hydrogen, Grade 5

6.9 Refrigerator capable of maintaining 4 °C

6.10 Freezer capable of maintaining temperatures below -15 °C

6.11 Repipetter – solvent delivery Optifix

6.12 Repeater digital – Eppendorf with appropriate size Combitips

7 REAGENTS

7.1 Solvents

7.1.1 Acetone – pesticide quality or equivalent

7.1.2 Iso-octane -pesticide quality or equivalent

7.1.3 80% Iso-octane/20% acetone: Combine 3200 mL of iso-octane with 800 mL of acetone in a 4 L solvent bottle. Measure the volumes independently. Label appropriately and assign a 6 month expiration date.

7.2 Solid Reagents – not used in this method.

7.3 Acids and Bases – not used in this method.

7.4 Stock Standards

7.4.1 Primary Aroclor stock standards are purchased from vendors as certified solutions at concentrations of 1000 or 100 µg/ml (Table 4). Alternative concentrations of Aroclor stock standards can be utilized to accommodate specific project objectives.

7.4.2 Primary chlorinated pesticide stock standards purchased for utilization in ECCS SOP LAM-003, OC Pesticides by 8081A, may be used in this method if needed as a chromatographic reference standard. This method is not applicable for the analysis of the chlorinated pesticides.

7.5 Intermediate Standards - This section is not applicable to this method.

7.6 Calibration Standards

7.6.1 Calibration standards are prepared at ten concentrations by dilution of the primary stock standards (Section 7.4.1) in 80% iso-octane/20% acetone to create a calibration curve (Table 6-1). A set of calibration standards is created for each individual Aroclor as needed. After the individual calibration standards are prepared, the solutions are entered into LIMS and transferred to LIMS labeled 40 mL VOA vials for storage. An example of how a standard solution is prepared is provided below.

7.6.1.1 Level 10, (2.0/0.24 µg/mL) Using a 250 µl gas tight syringe aliquot 200 µL of Aroclor stock at 1000 µg/mL (Section 7.4.1) and using a 4.0 mL volumetric pipette, aliquot 4.0 mL of the Surrogate Spike Mix TCMX/DCBP 6.0 µg/ml (Section 7.7.) into a 100 mL volumetric flask. Dilute to volume with 80% iso-octane/20% acetone.

7.6.1.2 See Table 6-1 for instruction on creation of the calibration standards Level 1 through Level 10.

7.6.2 If soil samples undergo sulfuric acid cleanup, the calibration and continuing calibration standards must also be acid treated.

7.7 Surrogate Spike

7.7.1 The surrogate spike mix (DCBP and TCMX at 6.0 µg/mL) is purchased from Absolute Standards (Absolute Part # 95677) in 1000 mL or 2000 mL batches.

7.7.2 No further dilution is required.

7.8 Laboratory Control Sample (LCS) Spike

7.8.1 One of the four primarily detected Aroclors (1242, 1248, 1254, and 1260) is typically used for LCs and MS/MSD spiking, usually the dominant Aroclor expected to be detected and fully calibrated. The spiking solutions are purchased from Absolute Standards at 100 µg/mL in acetone. Absolute part numbers are as follows:

Aroclor	**Absolute Part #.**
Aroclor 1242	94034
Aroclor 1248	94033
Aroclor 1254	94032
Aroclor 1260	95894

7.8.2 The stock spiking standards require no further dilution.

7.9 Matrix Spike / Matrix Spike Duplicate (MS/MSD) Spike

7.9.1 The same spike solution used for the Laboratory Control Spike (Section 7.8.1) is used for the matrix spike / matrix spike duplicate. The concentrations of the MS/MSD spiking solutions are found in Table 7.

7.9.2 The stock spiking standards require no further dilution.

7.10 Second Source Calibration Standards

7.10.1 Second source Aroclor stock standards are purchased from Absolute Standards at 100 µg/mL and are the same as the LCS spike standards (Section 7.8.1). The vendor for the second source standards should be different than the vendor not used as the source of the primary stock standards.

7.10.2 Second source calibration check standard.

7.10.2.1 Second source standards are prepared at 0.50/0.06 µg/mL. Using a gas tight syringe, aliquot 1 mL of the second source Aroclor stock (100 µg/ml) and using a volumetric pipette, aliquot 2.0 mL of surrogate mix at 6.0 µg/mL (see Section 7.7.4) into a 200 mL volumetric flask. Dilute to volume with 80% iso-octane/20% acetone.

7.10.2.2 Enter the standard into LIMS and transfer the standard to a LIMS labeled 40 mL VOA vials, store in a freezer and assign a one year expiration date.

7.11 Internal Standard – Not applicable to this method.

8 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Water samples should be collected in 1 L amber glass bottles with Teflon lined caps. Water samples must be extracted within 7 days of collection.

8.2 Soil samples should be collected in 4 ounce or larger amber glass jars with Teflon lined caps. Soil samples must be extracted within 14 days of collection. Hold times may be increased for soils to 1 year as long as they are frozen within the 14 day hold time window.

8.3 Wipe samples should be collected in 4 ounce or larger amber glass jars with Teflon lined caps. Wipe samples must be extracted within 14 days of collection.

8.4 All samples should be stored on ice or refrigerated at 4 °C immediately after collection. In the laboratory, store samples at 4 °C and out of direct sunlight at all times.

8.5 Extracts are stored in the freezer and must be analyzed within 40 days of extraction.

9 PROCEDURE

9.1 Preparation of Samples – Choose the appropriate preparation method below.

9.1.1 Water samples

9.1.1.1 PRE-001, Separatory Funnel Extraction

9.1.1.2 PRE-002, Self-Contained Water Extraction

9.1.2 Soil/sediment samples

9.1.2.1 PRE-003, Micro-Scale Soil Extraction

9.1.3 Wipe Samples

9.1.3.1 PRE-006, Wipe Sample Extraction

9.1.4 Waste

9.1.4.1 PRE-007, Waste Dilution

9.2 Clean-up of Samples – Choose the appropriate clean-up method below.

9.2.1 PRE-004, Sulfuric Acid Clean-up

Note: If soil samples undergo sulfuric acid cleanup, the calibration and continuing calibration standards must also be acid treated.

9.2.2 PRE-005, Mercury Clean-up

9.3 Instrument Conditions

9.3.1 Gas Chromatograph #1: RTX 35 Column, 0.53mm X 30m, 0.5µm, or equivalent

Temperature Program	
Initial Temp:	110 °C
Initial Hold:	2.0 min
Initial Rate:	15 °C/min
Final Temp (1):	330 °C
Hold Time (1):	2.0 min
Injector Temp:	280 °C
Detector Temp:	300 °C
Carrier Gas:	Helium
Head Pressure:	~7 PSI
Make up Gas:	Nitrogen
Flow Rate:	~30 mL/min
Split Vent:	~30-35 mL/min (total flow after column flow rate is set).
Septum Purge:	< 1.0 mL/min
Equilibration Time	1.50 min
Oven Max	350° C
Splitless Valve	Splitless box not checked
On time	0.5 min
Off time	3.00 min

NOTE: Instrument conditions may be modified to achieve optimum chromatographic response or to meet project specific requirements.

9.3.2 Gas Chromatograph #2: RTX-1701 Column, 0.53mm X 30m, 0.5µm, or equivalent

Temperature Program	
Initial Temp:	140 °C
Initial Hold:	1.0 min
Initial Rate:	5 °C/min
Final Temp:	280 °C
Hold Time:	2 min
Injector Temp:	250 °C
Detector Temp:	300 °C
Carrier Gas:	Helium
Head Pressure:	~7 PSI
Make up Gas:	Nitrogen
Flow Rate:	~30 mL/min

Split Vent:	~28 mL/min
Septum Purge:	< 1.0 mL/min
Equilibration Time	1.50 min
Oven Max	285° C
Splitless Valve	Splitless box not checked
On time	0.5 min
Off time	3.00 min

NOTE: Final temp for RTX-1701 must not exceed 280 °C. Temperatures above 280 °C cause rapid column degradation.

NOTE: Instrument conditions may be modified to achieve optimum chromatographic response or to meet project specific requirements. All instrument conditions other than those listed in this SOP must be documented.

NOTE: The chromatograms in Figures 1 through 7 portray examples of acceptable peak shape, peak resolution, compound response, and compound response ratios that are desired for each Aroclor..

9.4 Preventive Maintenance

9.4.1 Routine maintenance consists of clipping the column, replacing the liner and seal, cleaning the hat and installing a new septum.

9.4.2 Changing only the septum is acceptable on a daily basis or if a limited number of injections had been made on the GC.

9.4.3 Carryover on the GC is usually caused by a worn out injection syringe. When replacing the syringe, use fine sandpaper to file off the sharp point and/or burr. Rinse with solvent from the top to remove any grit left in the syringe tip.

9.4.4 The need for preventive maintenance in improving chromatographic response can only be determined by sufficient training and experience in the analysis of Aroclors. As stated in section 1.7, this method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and skilled in the interpretation of chromatograms.

9.5 Calibration

9.5.1 If soil samples undergo sulfuric acid cleanup, the calibration and continuing calibration standards must also be acid treated.

9.5.2 For one of the primary Aroclors of interest, prepare an initial calibration curve with a minimum of 5 concentrations from the ten available (see Table 6). Aroclor 1242, 1248, 1254, or 1260 are usually used for the multipoint ICAL. One of the standards in the curve must be at or below the limit of quantitation. .

9.5.3 Appropriate single point calibration standards at 0.5 µg/mL must also be analyzed during the initial calibration that include at a minimum, 1016 and 1260 to encompass the full Aroclor retention time range.

9.5.4 The example chromatograms in Figures 1 through 7 provide target peaks that are typically used for calibration and quantitation. Other peaks may be selected based on the individual chromatogram, but the targets must be attempted first.

9.5.5 Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph. Refer to Table 8 for a typical sequence. Plot the peak height responses against the concentration of the calibration standard using power regression. The coefficient of determination (r^2) must be ≥0.990, which corresponds to a correlation coefficient of (r) of ≥0.995.

NOTE: Peak heights are normally used for all calculations.

$$Regression\text{:}\ Y = AX^B\ or\ \ln Y = B\ln X + \ln A$$

Where: Y = Peak height
X = Concentration in µg/mL
A = Constant
B = Exponent

9.5.6 Continuing calibration verification (CCV) -The working calibration curve is verified on each working day by the injection of one or more continuing calibration verification standards. If the response for any Aroclor varies from theory by more than ± 20%, a new calibration curve must be injected, and/or data are qualified.

$$Percent\ Difference = \frac{R2 - R1}{R1} \times 100$$

Where: R1 = Theoretical concentration.
R2 = Concentration from CCV.

9.6 Retention Time Windows

9.6.1 Retention times obtained from the ICAL are used to update the calibration file.

9.6.2 The ICAL retention times once established may be adjusted as processing the data occurs using CCVs, LCSs and MS/MSDs for reference retention times. Normally windows are established for the surrogate TCMX/DCBP at approximately 0.10 minutes. The surrogates are set as reference peaks, and as such the rest of the retention times will shift with the surrogate on an injection-by-injection basis.

9.6.3 The retention windows for the Aroclor target peaks are usually set between 0.04 and 0.06 minutes.

9.7 Sample Analysis

9.7.1 Set up the GC system using the conditions described in Section 9.3.

9.7.2 Samples are analyzed in a set referred to as a LIMS run sequence. See Table 8 for an example of a typical sequence. CCVs must be injected every 10 samples or less and at the end of the run. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded. Raw data is collected and archived by the LIMS sequence number. .

9.7.3 Aliquots of the standards, samples and QC are transferred using disposable pipets into properly labeled injection vials.

NOTE: The use of 250 µL inserts in the injection vials to preserve standard or sample extract volume is recommended.

Note: If soil samples undergo sulfuric acid cleanup, the calibration and continuing calibration standards must also be acid treated.

9.7.4 Injection – Inject approximately 2 µL of the sample extract. The data system will record the resulting peak height for each peak of interest.

9.7.5 If the calculated value exceeds the theoretical value of the highest standard, dilute the extract and re-inject. Dilute the sample so that all peaks are on scale but in the upper half of the standard curve range.

9.7.6 If target peak detection is prevented by the presence of interferences, further cleanup may be required (Refer to cleanup SOPs PRE-004 and PRE-005). If an interference is present, a target peak(s) may be eliminated from the number of peaks used in the total for that particular Aroclor.

9.7.7 Qualitative identification of Aroclors in a sample is based on pattern recognition of the Aroclor standard versus the pattern in the sample, if present. This identification is based on professional judgment.

NOTE: If multiple Aroclors are present in a sample causing target peak co-elution, the analyst must use professional judgment whether to include or exclude specific target peaks from the calculation of each Aroclor.

9.7.8 Quantitation of unknown samples is done by summing the results of the regression analysis for each target peak. Each of the target peaks identified by retention time is calculated by the data system to obtain a µg/mL value. To determine the amount of Aroclor, in µg/mL, in each sample, the µg/mL values for each target peak are added together and then divided by the total number of target peaks used.

9.7.9 There are several possible interferences that may occur, which prevent accurate calculation of some peaks, (i.e. sulfur, DDT and metabolites, unknown interference not removed by concentrated H_2SO_4, technical chlordane, toxaphene, 8081 pesticides). Any of these may result in either a peak that is buried under a large peak or one or more peaks that have elevated results compared to the rest of the positive hits. If any of the above occur, the peaks that are interfered with are excluded from the total calculation resulting in the division number being smaller than 9. (i.e. if peaks 2 and 5 are elevated, these peak concentrations are not included in the total concentrations and the divisor would be 7.

9.7.10 Chromatography Data Assembly and Review - Chromatography data are to be assembled and reviewed in accordance with ECCS SOP GEN-016, Data Review.

9.8 Calculations

9.8.1 The concentration of each Aroclor in the sample extract is determined from the calibration curve in µg/ml through reverse extrapolation from the peak height response in the sample using power regression. The concentration in µg/L or µg/kg of each of the Aroclors in the sample is then calculated as follows:

9.8.1.1 Water Samples (µg/L)

$$Concentration\ (\mu g/L) = \frac{A_x \times D \times V_e}{V_s}$$

Where: A_x = Concentration of Aroclor in the extract in µg/mL
D = Dilution factor, if applicable
V_e = Volume of extract in mL
V_s = Volume of sample extracted in liters

9.8.1.2 Soil Samples (µg/kg)

$$Concentration\ (\mu g/g) = \frac{A_x \times D \times V_e}{W_s}$$

Where: A_x = Concentration of Aroclor in the extract in µg/mL
D = Dilution factor, if applicable
V_e = Volume of extract in mL
W_s = Volume of sample extracted in kilograms

9.8.1.3 Wipe Samples (µg)

9.8.1.3.1 Wipe samples are reported in total micrograms. The total microgram result can be converted into a weight/area result if the area is supplied by the client. The calculation is performed as follows:

$$\text{Concentration } (\mu g) = A_x \times D \times V_e$$

Where: A_x = Concentration of Aroclor in the extract in µg/mL
D = Dilution factor, if applicable
V_e = Volume of extract in mL

10 QUALITY CONTROL

10.1 Refer to Method 8000 for general quality control procedures for chromatography methods.

10.2 An analytical batch consists of 20 or fewer samples. Batch quality control samples should be analyzed with each set with the following frequency:

Blanks - One per 20 or fewer samples, minimum one per day
LCSs - One per 20 or fewer samples, minimum one per day
MS/MSDs - One MS/MSD per 20 or fewer samples, minimum one set per day

If an MS/MSD cannot be prepared because of limited sample volume, a second LCS must be prepared.

10.3 Method blanks consist of an aliquot of laboratory reagent water or silica sand that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. If target analytes or interferences are present at concentrations that impact the analytical results for samples, the samples (including quality control samples) should be re-extracted or appropriately qualified in accordance with GEN-015, Qualification of Data.

10.4 LCSs consist of an aliquot of laboratory reagent water or silica sand spiked with the target analytes, prepared and processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. LCS control limits for precision and accuracy are established on at least a yearly basis through the use of at least 20 data points. For Aroclors with insufficient data to calculate control limits, the control limit shall be 70-130%. If the recovery of any of the target Aroclor is outside control limits, the samples (including quality control samples) should be re-extracted or appropriately qualified in accordance with GEN-015, Qualification of Data.

10.5 MS/MSD samples consist of duplicate aliquots of sample spiked with the target analytes, prepared and processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. MS/MSD control limits for precision and accuracy are established on at least a yearly basis through the use of at least 20 data points. For Aroclors with insufficient data to calculate control limits, the control limit shall be 60-140% and 20% RPD. MS/MSD control limits are advisory. If the recovery or RPD of any of the target analytes is outside control limits, data should be appropriately qualified in accordance with GEN-015, Qualification of Data.

10.6 Initial calibration (ICAL) is performed using the external standard technique by injecting a minimum of 5 of the available calibration standards. The lowest

calibration standard must be at or below the limit of quantitation. The coefficient of determination (r^2) must be ≥0.990, which corresponds to a correlation coefficient of (r) of ≥0.995.

10.7 The working calibration curve must be verified on each working day by the injection of one or more CCV standards. If the response for any Aroclor varies from the theoretical concentration by more than 20%, a new calibration curve must be prepared or data appropriately qualified in accordance with GEN-015, Qualification of Data.

10.8 Surrogates are added to every sample and QC sample. Surrogate control limits are generated on at least a yearly basis. If a surrogate recovery is outside of control limits, the sample should be re-extracted and re-analyzed, if possible. If not, the data should be appropriately qualified in accordance with GEN-015, Qualification of Data.

10.9 A second source calibration verification standard must be analyzed with every initial calibration. The accepted limits are 70-130% for all Aroclors. If an SCV fails, immediate corrective action is required before proceeding with sample analysis. Affected data should be qualified according to GEN-015, Qualification of Data.

11 METHOD PERFORMANCE

11.1 Estimated limits of detection (LODs) for eight replicates of laboratory reagent water, silica sand, and wipes are listed in Table 1.

11.2 Typical demonstration of capability (DOC) data for laboratory reagent water and silica sand are summarized in Table 2.

12 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

12.1 Contingencies for out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and filed with the Quality Manager. Analytical results shall be qualified as necessary.

13 WASTE MANAGEMENT / POLLUTION PREVENTION

13.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

14 REFERENCES

14.1 This is an ECCS procedure. The technical elements and procedural requirements of the following methods were considered in preparation of this SOP.

14.1.1 SW 846, Method 8082, Revision 0, December 1996.

TABLE 1-1

LIMIT OF DETECTION
PCBS AS AROCLORS

Soil

	Mean Conc. (µg/kg)	Avg. % Rec. (µg/kg)	% RSD	LOD (µg/kg)
Aroclor 1016[1]	85.0	84.9	3.1	7.8
Aroclor 1221[1]	0.0212	106	6.2	4.1
Aroclor 1232[1]	0.0221	111	11	7.0
Aroclor 1242[2]	0.0201	101	7.3	4.4
Aroclor 1248[2]	0.024	118	4.1	2.9
Aroclor 1254[2]	0.021	105	4.7	3.0
Aroclor 1260[2]	0.021	107	4.3	2.8

[1]Data Source: A0L1302 December 17, 2010
[2]Data Source: A1A0601 January 10, 2011

Water (Self-contained Extraction)

	Mean Conc. (µg/L)	Avg. % Rec. (µg/L)	%RSD	LOD (µg/L)
Aroclor 1016	NA	NA	NA	NA
Aroclor 1221[1]	0.17	132	4.0	0.020
Aroclor 1232[1]	0.16	128	7.6	0.037
Aroclor 1242[2]	0.16	125	7.1	0.033
Aroclor 1248[2]	0.13	101	5.2	0.020
Aroclor 1254[2]	0.12	99.3	2.4	0.009
Aroclor 1260[2]	0.128	102	6.4	0.025

[1]DATA SOURCE: A0L1302 DECEMBER 17, 2010
[2]Data Source: A1A1301 January 13, 2011

Soil-Acid Treated

	Mean Conc. (µg/kg)	Avg. % Rec. (µg/kg)	% RSD	LOD Treated (µg/kg)
Aroclor 1016[1]				
Aroclor 1221[1]	0.0218	109	9.6	6.3
Aroclor 1232[1]	0.0222	111	4.2	4.2
Aroclor 1242[2]	0.0228	114	6.5	4.4
Aroclor 1248[2]				
Aroclor 1254[2]				
Aroclor 1260[2]				

[1]Data Source: A0L1302 December 17, 2010
[2]Data Source: A1A0601 January 10, 2011

TABLE 2

TYPICAL DEMOSTRATION OF CAPABILITY DATA

WATER – SELF CONTAINED

Compound	Spike Level (µg/L)	Mean Recovery	Avg. % Recovery	%RSD
Aroclor 1254	12.5	13.8	110	3.1
DCBP	-	0.79	106	1.0
TCMX	-	0.66	88.0	5.0

Data Source: A1A1301 January 14, 2011

TABLE 3-1

TYPICAL RETENTION TIME (IN MINUTES) OF AROCLORS BY GC

The peaks selected as quantitation peaks for Aroclor 1260, Aroclor 1254, Aroclor1248 and Aroclor 1242 are listed in reports and chromatograms in Figures 1.1 through 4.2. These are used for the analysis of these Aroclors. If sample chromatogram and multiple Aroclor presence indicate that alternative peaks should be used as quantitation peaks, then peak substitution is allowable.

The peaks selected as quantitation peaks for Aroclor 1016, Aroclor 1221, and Aroclor 1232 are listed in reports and chromatograms in Figures 5.1 through 7.2. These peaks are not defined as the quantitation peaks but are listed merely as examples of peak selection and Aroclor chromatography patterns.

TYPICAL RETENTION TIME (IN MINUTES) OF CHLORINATED PESTICIDES BY GC
MIX 1

Compound	**RTX-35**	**RTX-1701**
TCMX (surrogate)	7.386	7.791
α-BHC	9.292	11.168
γ-BHC (Lindane)	10.365	12.561
β-BHC	10.596	15.538
Heptachlor	11.430	13.097
δ-BHC	11.558	16.051
Aldrin	12.314	13.888
Heptachlor epoxide	13.764	16.295
γ-Chlordane	14.267	17.404
α-Chlordane	14.687	17.612
Endosulfan I	15.254	17.051
4,4’-DDE	15.468	18.046
Dieldrin	16.331	18.834
Endrin	16.632	18.834
4,4’-DDD	16.790	20.574
Endosulfan II (1)	17.409	20.478
4,4’-DDT (1)	17.409	21.003
Endrin aldehyde	17.542	21.812
Endosulfan sulfate	17.944	22.825
Methoxychlor	19.357	23.080
Endrin ketone	19.681	23.837
DCBP (Surrogate)	22.687	26.528

(1) Co-elute on RTX-35

TABLE 3-2

TYPICAL RETENTION TIMES (IN MINUTES) OF CHLORINATED PESTICIDES BY GC MIX 2

Compound	**RTX-35**	**RTX-1701**
Hexachlorobenzene	8.881	9.457
Pentachlorobenzene	10.077	11.594
2,4'-DDE	14.583	16.970
Trans – Nonachlor	14.422	17.732
2,4'-DDD	15.824	19.059
2,4'-DDT	16.614	19.395
Cis –Nonachlor	16.497	20.604
Mirex	19.863	21.880

TABLE 4

STOCK STANDARD CONCENTRATIONS

PRIMARY AROCLOR STOCK STANDARD CONCENTRATION

Target Mixture	**µg/mL**
Aroclor 1016	1000
Aroclor 1221	1000
Aroclor 1232	1000
Aroclor 1242	1000
Aroclor 1248	1000
Aroclor 1254	1000
Aroclor 1260	1000

SECOND SOURCE AROCLOR STOCK STANDARD CONCENTRATION

Target Mixture	**µg/mL**
Aroclor 1016	1000
Aroclor 1221	1000
Aroclor 1232	1000
Aroclor 1242	100
Aroclor 1248	100
Aroclor 1254	100
Aroclor 1260	100

TABLE 5

INTERMEDIATE STANDARD CONCENTRATIONS

Not applicable to this method.

TABLE 6-1

PREPARATION OF AROCLOR INITIAL CALIBRATION STANDARDS

CONCENTRATION (µg/mL)

Target Mixture / Compound	**L-1**	**L-2**	**L-3**	**L-4**	**L-5**	**L-6**	**L-7**	**L-8**	**L-9**	**L-10**
Aroclor Std.	0.002	0.005	0.01	0.02	0.05	0.10	0.20	0.5	1.0	2.0[1]
TCMX (surrogate)	0.00024	0.006	0.0012	0.0024	0.006	0.012	0.024	0.06	0.12	0.24
DCBP (Surrogate)	0.00024	0.006	0.0012	0.0024	0.006	0.012	0.024	0.06	0.12	0.24

DILUTION VOLUMES FOR AROCLOR CALIBRATION CURVES

	L-1	**L-2**	**L-3**	**L-4**	**L-5**	**L-6**	**L-7**	**L-8**	**L-9**	**L-10**
Aliquot (mL) [2]	10	10	10	10	10	10	10	0.10	0.10	0.20
Stock ID µg/mL	-	-	-	-	-	-	-	1000	1000	1000
Level ID	L-4	L-5	L-6	L-7	L-8	L-9	L-10	-	-	-
Aliquot (mL) [3]	10	10	10	10	10	10	10	2.0	2.0	4.0
Surrogate ID µg/mL	-	-	-	-	-	-	-	6.0	6.0	6.0
Surrogate ID	L-4	L-5	L-6	L-7	L-8	L-9	L-10	-	-	-
Final Volume mL[4]	100	100	100	100	100	100	100	200	100	100

NOTE:
[1] Calibration standards greater than 2.0 µg/mL may be prepared as long as acceptable standard curves are obtained.
[2] Primary Aroclor stock standard (Section 7.4.1)
[3] Surrogate spike mix (Section 7.7.4)
[4] Solvent: 80% iso-octane/20% acetone

TABLE 7

CONCENTRATION OF LCS AND MS/MSD SPIKE SOLUTION

Aroclor	**Concentration (µg/mL)**
All	100 µg/ml

Note: Aroclors 1242, 1248, 1254 and 1260 are usually used for LCS and MS/MSD.

TABLE 8

ICAL RUN SEQUENCE ORDER

Description	**Concentration (µg/mL)**
Aroclor 1221	0.05
Aroclor 1016	0.05
Aroclor 1232	0.05
Aroclor 1242	0.05
Aroclor 1248	0.05
Aroclor 1260	0.05
Aroclor 1254[1]	0.002
Aroclor 1254[1]	0.005
Aroclor 1254[1]	0.01
Aroclor 1254[1]	0.02
Aroclor 1254[1]	0.05
Aroclor 1254[1]	0.1
Aroclor 1254[1]	0.2
Aroclor 1254[1]	0.5
Aroclor 1254[1]	1.0
Aroclor 1254[1]	2.0
Blank 80/20	-
Second Source 1242	0.5
Second Source 1248	0.5
Second Source 1254	0.5
Second Source 1260	0.5
ICV 1242	0.5
ICV 1248	0.5
ICV 1254	0.5
ICV 1260	0.5

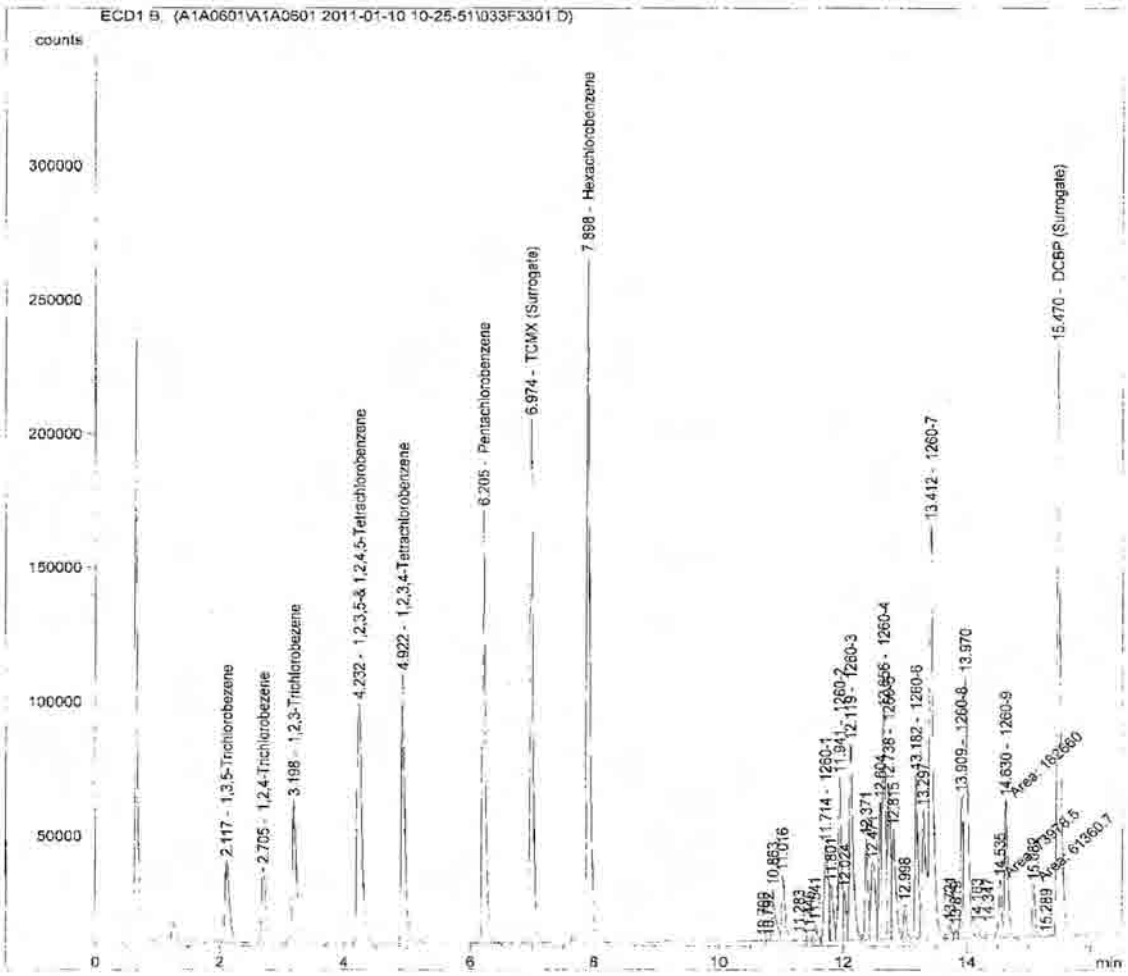
[1]Any of the Aroclors could be fully calibrated based on project specific needs.

FIGURE 1.1

CHROMATOGRAM OF AROCLOR 1260 & CHLORINATED BENZENES

Data File C:\CHEM32\4\DATA\A1A0601\A1A0601 2011-01-10 10-25-51\033F3301.D
Sample Name: A1A0601-CALS

Acq. Operator	: CJM	Seq. Line	: 33
Acq. Instrument	: 3310A49834	Location	: Vial 33
Injection Date	: 1/10/2011 11:29:50 PM	Inj	: 1
		Inj Volume	: Manually
Acq. Method	: C:\CHEM32\4\DATA\A1A0601\A1A0601 2011-01-10 10-25-51\PCB.M		
Last changed	: 12/1/2010 3:51:50 PM by RBO		
Analysis Method	: C:\CHEM32\4\METHODS\60CBA1A0601.M		
Last changed	: 6/28/2011 5:36:37 PM by cjm (modified after loading)		
Method Info	: Method 8082. Column Restek Rtx-35 30 M X 0.53 mm X 0.5 um Film S/N 976125.		



3310A49834 6/28/2011 5:38:28 PM cjm

Page 1 of 3

FIGURE 1.2

REPORT OF AROCLOR 1260

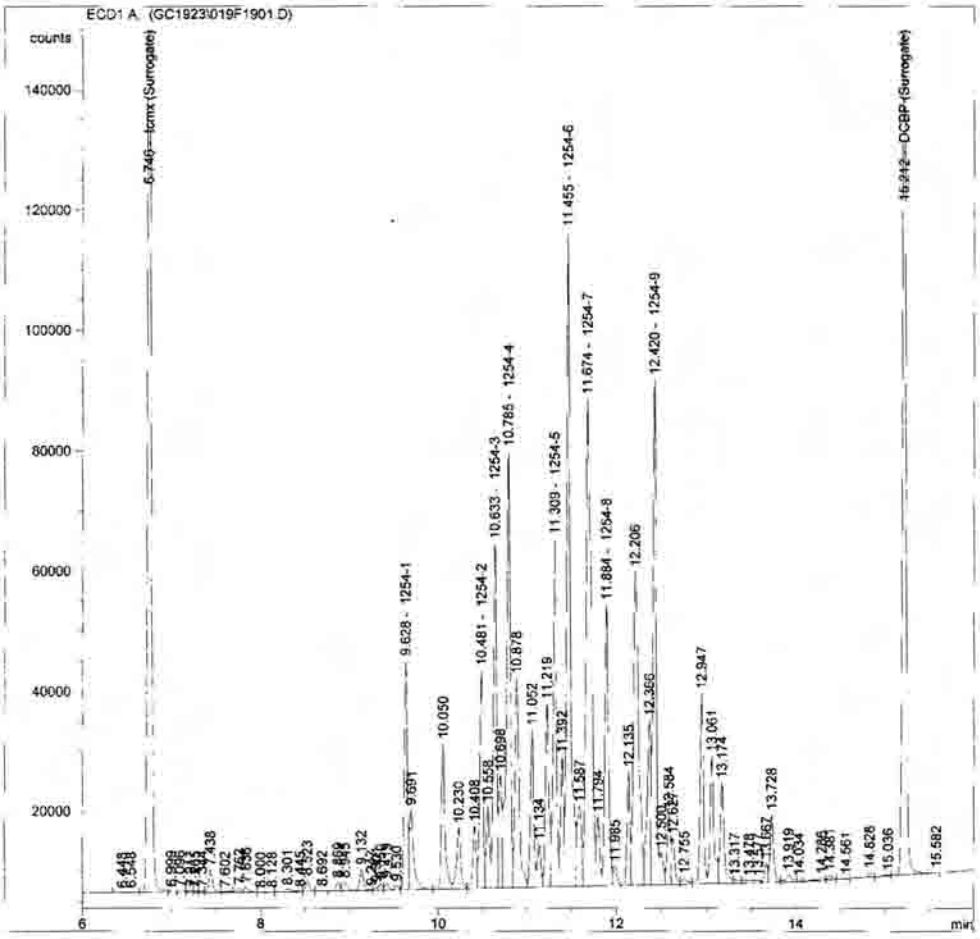
```
Data File C:\CHEM32\4\DATA\A1A0601\A1A0601 2011-01-10 10-25-51\033F3301.D
Sample Name: A1A0601-CALS
==========================================================================
Acq. Operator    : CJM                          Seq. Line :  33
Acq. Instrument  : 3310A49834                    Location : Vial 33
Injection Date   : 1/10/2011 11:29:50 PM              Inj :   1
                                               Inj Volume : Manually
Acq. Method      : C:\CHEM32\4\DATA\A1A0601\A1A0601 2011-01-10 10-25-51\PCB.M
Last changed     : 12/1/2010 3:51:50 PM by RBO
Analysis Method  : C:\CHEM32\4\METHODS\60CBA1A0601.M
Last changed     : 6/28/2011 5:36:37 PM by cjm
                   (modified after loading)
Method Info      : Method 8082.  Column Restek Rtx-35  30 M  X  0.53 mm  X  0.5 um Film  S/
                   N  976125.

==========================================================================

==========================================================================
                        External Standard Report
==========================================================================

Sorted By              :       Retention Time
Calib. Data Modified   :       1/12/2011 11:19:47 AM
Multiplier:                     :       1.0000
Dilution:                       :       1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: ECD1 B,

RetTime Sig Type    Height    Amt/Height   Amount   Grp   Name
 [min]              [counts]                [UG/ML]
-------|--|------|----------|----------|----------|--|--------------
  2.117  1 VV     3.07717e4  1.76044e-6 5.41718e-2     1,3,5-Trichlorobezene
  2.705  1 VV     2.82224e4  1.88967e-6 5.33312e-2     1,2,4-Trichlorobezene
  3.198  1 VV     5.34840e4  9.94810e-7 5.32064e-2     1,2,3-Trichlorobezene
  4.232  1 VV     8.99385e4  1.14566e-6 1.03039e-1     1,2,3,5-& 1,2,4,5-Tetrachlorobenzene
  4.922  1 VV     1.00407e5  5.14202e-7 5.16297e-2     1,2,3,4-Tetrachlorobenzene
  6.205  1 VV     1.61875e5  3.12441e-7 5.05764e-2     Pentachlorobenzene
  6.974  1 VV  +  1.95757e5  3.15404e-7 6.17426e-2     TCMX (Surrogate)
  7.898  1 BB     2.56468e5  1.98058e-7 5.07955e-2     Hexachlorobenzene
 11.714  1 VV     3.56585e4  1.39917e-5 4.98923e-1  1 1260-1
 11.941  1 VV     6.10714e4  8.18626e-6 4.99947e-1  1 1260-2
 12.119  1 VV     7.34947e4  6.70686e-6 4.92919e-1  1 1260-3
 12.656  1 VV     8.55216e4  5.98078e-6 5.11486e-1  1 1260-4
 12.738  1 VV     5.76979e4  9.40089e-6 5.42412e-1  1 1260-5
 13.182  1 VV     6.17043e4  8.22350e-6 5.07425e-1  1 1260-6
 13.412  1 VV     1.54598e5  3.28203e-6 5.07395e-1  1 1260-7
 13.909  1 VV     5.32726e4  9.55548e-6 5.09045e-1  1 1260-8
 14.630  1 FM     5.14327e4  1.00382e-5 5.16291e-1  1 1260-9
 15.470  1 BB  +  2.20580e5  2.68993e-7 5.93343e-2     DCBP (Surrogate)

Totals :                                  5.12367


Group summary :

 Group  Use   Height      Amount       Group Name
  ID         [counts]    [UG/ML]
|------|---|----------|----------|-------------------------|
  1         6.34452e5     4.58584  AROCLOR 1260

1 Warnings or Errors :

3310A49834 6/28/2011 5:38:28 PM cjm                                    Page   2 of 3
```

FIGURE 2.1

CHROMATOGRAM OF AROCLOR 1254

Data File D:\HPCHEM\2\DATA\GC1923\019F1901.D Sample Name: CCV 1254 0.5
LOG 173 PAGE 85

```
==================================================================
Injection Date  : 2/7/2008 10:19:25 PM          Seq. Line :  19
Sample Name     : CCV 1254 0.5                       Vial :  19
Acq. Operator   : FUZ                                 Inj :   1
                                                Inj Volume : Manually
Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
Last changed    : 2/7/2008 2:46:21 PM by FUZ
Analysis Method : D:\HPCHEM\2\METHODS\54-1923.M
Last changed    : 3/24/2008 10:44:06 AM by RBO
                  (modified after loading)
FIELD PCB'S BY 8082. COLUMN RESTEK RTX-35 30M X 0.53 ID X 0.5 MICRON FILN S/
N (720902). 2uL injection splitless
==================================================================
```



3203A41034 3/24/2008 10:44:06 AM RBO Page 1 of 2

FIGURE 2.2

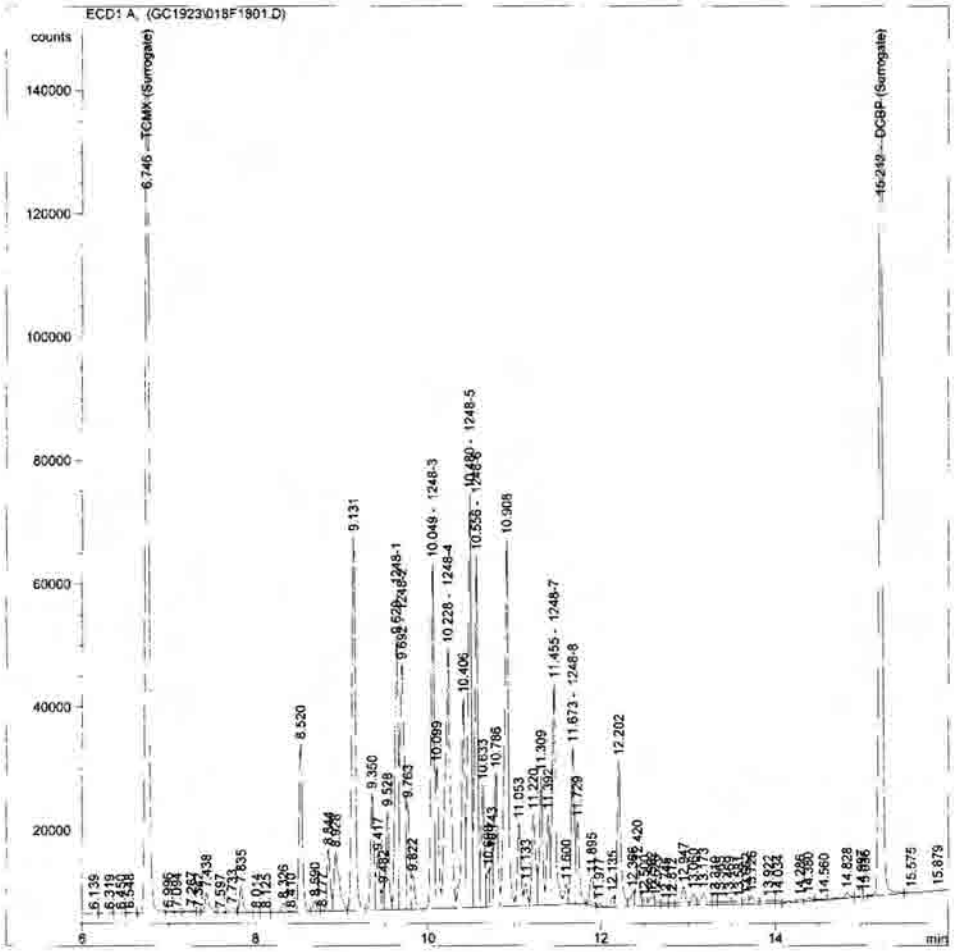
REPORT OF AROCLOR 1254

```
Data File D:\HPCHEM\2\DATA\GC1923\019F1901.D          Sample Name: CCV 1254 0.5
    LOG 173 PAGE 85

    ==================================================================
    Injection Date  : 2/7/2008 10:19:25 PM          Seq. Line :  19
    Sample Name     : CCV 1254 0.5                       Vial :  19
    Acq. Operator   : FUZ                                 Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\54-1923.M
    Last changed    : 3/24/2008 10:44:06 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8082. COLUMN RESTEK RTX-35 30M X 0.53 ID X 0.5 MICRON FILN S/
    N (720902). 2uL injection splitless
    ==================================================================

    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By              :      Retention Time
    Calib. Data Modified   :      2/9/2008 2:35:06 PM
    Multiplier             :      1.0000
    Dilution               :      1.0000

    Signal 1: ECD1 A,

    RetTime Sig Type    Height    Amt/Height   Amount   Grp   Name
     [min]             [counts]                [ug/ml]
    -------|--|------|----------|----------|----------|--|---------------
      6.746  1 VV  +  2.23489e5  2.68469e-7 6.00000e-2  1 tcmx (Surrogate)
      9.628  1 VV     3.81552e4  1.31044e-5 5.00000e-1  2 1254-1
     10.481  1 VV     3.62775e4  1.37826e-5 5.00000e-1  2 1254-2
     10.633  1 VV     5.73636e4  8.71634e-6 5.00000e-1  2 1254-3
     10.785  1 VV     7.26741e4  6.88003e-6 5.00000e-1  2 1254-4
     11.309  1 VV     5.79228e4  8.63218e-6 5.00000e-1  2 1254-5
     11.455  1 VV     1.08978e5  4.58810e-6 5.00000e-1  2 1254-6
     11.674  1 VV     8.09502e4  6.17664e-6 5.00000e-1  2 1254-7
     11.884  1 VV     4.67519e4  1.06947e-5 5.00000e-1  2 1254-8
     12.420  1 VV     8.40903e4  5.94599e-6 5.00000e-1  2 1254-9
     15.212  1 VV  +  1.90063e5  3.15685e-7 6.00000e-2  1 DCBP (Surrogate)

    Totals :                                 4.71912e5

     Results obtained with standard integrator!
    Group summary :

     Group  Use   Height      Amount       Group Name
      ID         [counts]    [ug/ml]
    |------|---|----------|----------|-------------------------|
      1         4.13552e5  1.20000e-1  Surrogate
      2         5.83163e5     4.50000  Aroclor-1254

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    ==================================================================
                            *** End of Report ***

3203A41034 3/24/2008 10:44:06 AM RBO                      Page 2 of 2
```

FIGURE 3.1

CHROMATOGRAM OF AROCLOR 1248

```
Data File D:\HPCHEM\2\DATA\GC1923\018F1801.D          Sample Name: CCV 1248 0.5
    ==================================================================
    Injection Date  : 2/7/2008 9:54:21 PM            Seq. Line :  18
    Sample Name     : CCV 1248 0.5                        Vial :  18
    Acq. Operator   : FUZ                                  Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\48-1923.M
    Last changed    : 3/24/2008 10:23:08 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8082. COLUMN RESTEK RTX-35 30M X 0.53 ID X 0.5 MICRON FILN S/
    N (482893). 2uL  injection
    ==================================================================
```



3203A41034 3/24/2008 10:23:09 AM RBO Page 1 of 2

FIGURE 3.2

REPORT OF AROCLOR 1248

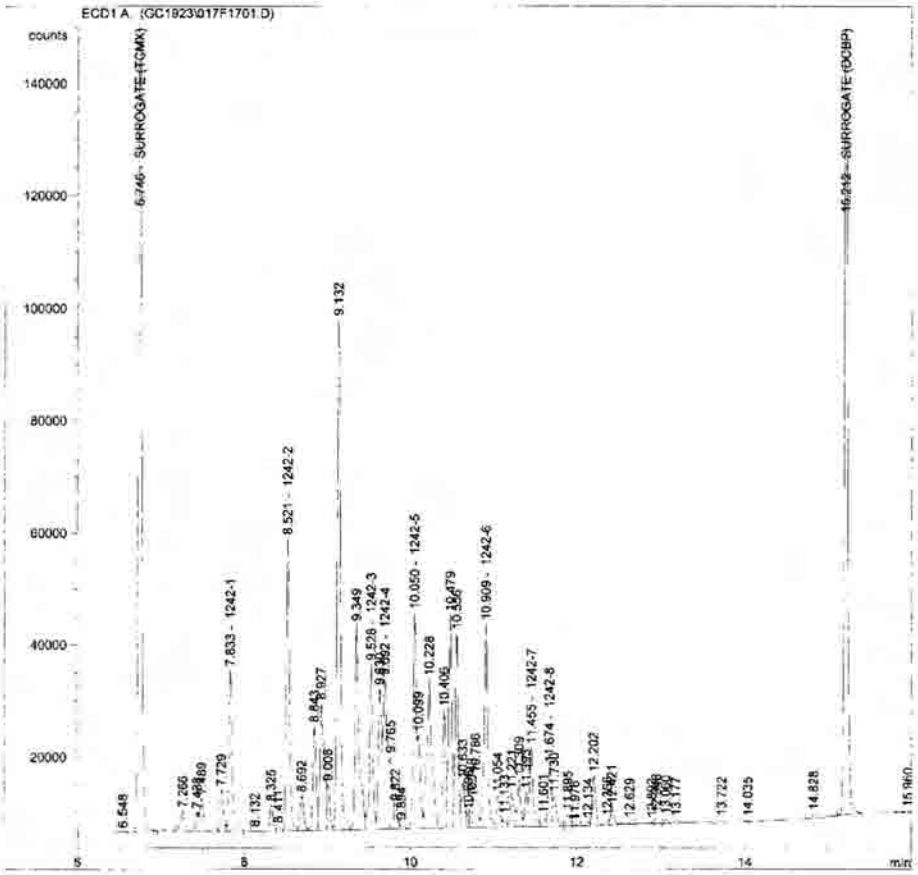
```
Data File D:\HPCHEM\2\DATA\GC1923\018F1801.D          Sample Name: CCV 1248 0.5
    ==================================================================
    Injection Date  : 2/7/2008 9:54:21 PM            Seq. Line :  18
    Sample Name     : CCV 1248 0.5                        Vial :  18
    Acq. Operator   : FUZ                                  Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\48-1923.M
    Last changed    : 3/24/2008 10:23:08 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8082. COLUMN RESTEK RTX-35 30M X 0.53 ID X 0.5 MICRON FILN S/
    N (482893). 2uL  injection
    ==================================================================

    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By              :      Retention Time
    Calib. Data Modified   :      2/8/2008 6:31:02 PM
    Multiplier             :      1.0000
    Dilution               :      1.0000

    Signal 1: ECD1 A,

    RetTime Sig Type    Height   Amt/Height   Amount   Grp   Name
     [min]             [counts]                [ug/ml]
    -------|--|------|----------|----------|----------|--|---------------
      6.746  1 VV  +  2.06751e5  2.88197e-7 5.95850e-2  1 TCMX (Surrogate)
      9.629  1 VV     4.40319e4  1.12558e-5 4.95616e-1  2 1248-1
      9.692  1 VV     3.98310e4  1.23261e-5 4.90960e-1  2 1248-2
     10.049  1 PV     5.62141e4  8.79145e-6 4.94203e-1  2 1248-3
     10.228  1 VV     4.23060e4  1.18305e-5 5.00500e-1  2 1248-4
     10.480  1 VV     6.72533e4  7.38270e-6 4.96510e-1  2 1248-5
     10.556  1 VV     5.71933e4  8.72139e-6 4.98805e-1  2 1248-6
     11.455  1 VV     3.59946e4  1.39880e-5 5.03493e-1  2 1248-7
     11.673  1 VV     2.55046e4  1.98998e-5 5.07535e-1  2 1248-8
     15.212  1 VV  +  1.91884e5  3.14773e-7 6.03998e-2  1 DCBP (Surrogate)

    Totals :                                 5.06455e5

     Results obtained with standard integrator!
    Group summary :

     Group  Use   Height     Amount      Group Name
      ID         [counts]    [ug/ml]
    |------|---|----------|----------|--------------------------|
      1         3.98635e5  1.19985e-1  Surrogate
      2         3.68329e5     3.98762  Aroclor-1248

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    ==================================================================
                            *** End of Report ***

3203A41034 3/24/2008 10:23:09 AM RBO                      Page 2 of 2
```

FIGURE 4.1

CHROMATOGRAM OF AROCLOR 1242

Data File D:\HPCHEM\2\DATA\GC1923\017F1701.D Sample Name: CCV 1242 0.5
LOG 173 PAGE 89

```
==================================================================
Injection Date  : 2/7/2008 9:29:19 PM          Seq. Line :  17
Sample Name     : CCV 1242 0.5                      Vial :  17
Acq. Operator   : FUZ                                Inj :   1
                                              Inj Volume : Manually
Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
Last changed    : 2/7/2008 2:46:21 PM by FUZ
Analysis Method : D:\HPCHEM\2\METHODS\42-1923.M
Last changed    : 3/24/2008 10:18:23 AM by RBO
                  (modified after loading)
FIELD PCB'S BY 8081. COLUMN RESTEK RTX-35 30M X 0.35 ID X 0.50 MICRON FILM
S/N 566535. 3ul injection with gooseneck liner.
==================================================================
```



3203A41034 3/24/2008 10:18:23 AM RBO Page 1 of 2

FIGURE 4.2

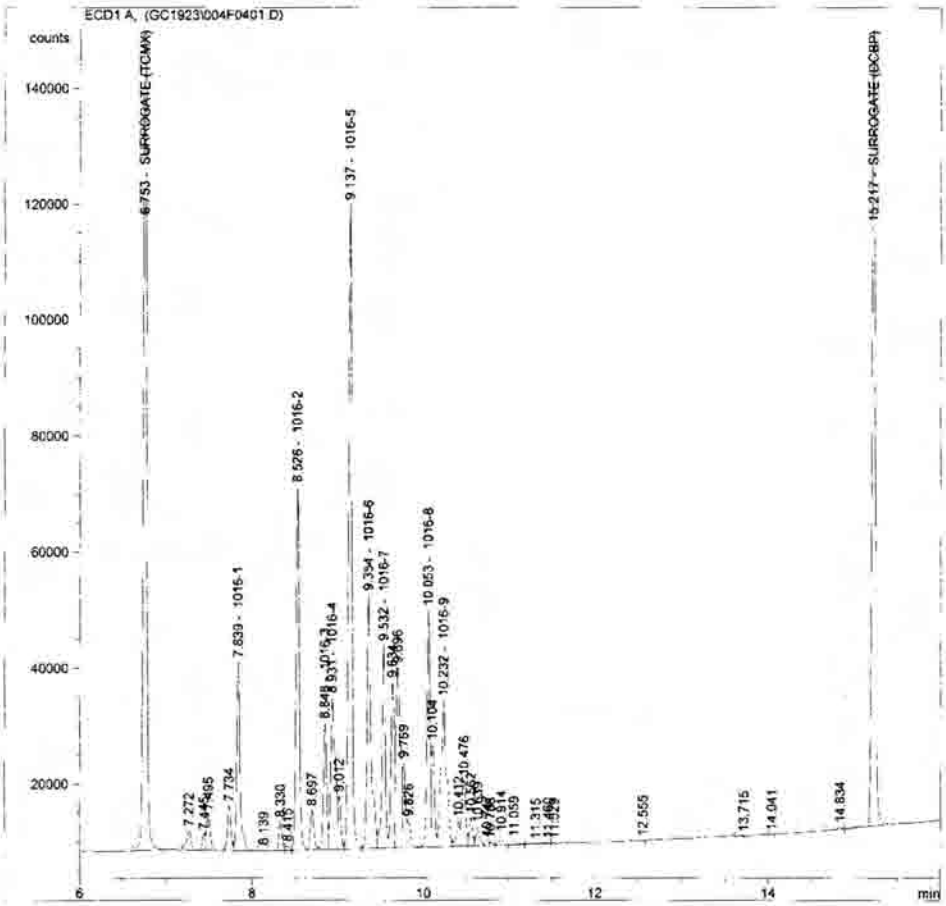
REPORT OF AROCLOR 1242

```
Data File D:\HPCHEM\2\DATA\GC1923\017F1701.D          Sample Name: CCV 1242 0.5
    LOG 173 PAGE 89

    ==================================================================
    Injection Date  : 2/7/2008 9:29:19 PM            Seq. Line :  17
    Sample Name     : CCV 1242 0.5                        Vial :  17
    Acq. Operator   : FUZ                                  Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\42-1923.M
    Last changed    : 3/24/2008 10:18:23 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8081. COLUMN RESTEK RTX-35 30M X 0.35 ID X 0.50 MICRON FILM
    S/N 566535. 3ul injection with gooseneck liner.
    ==================================================================

    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By              :      Retention Time
    Calib. Data Modified   :      2/9/2008 2:22:09 PM
    Multiplier             :      1.0000
    Dilution               :      1.0000
    Sample Amount          :      1.00000  [UG/ML]    (not used in calc.)

    Signal 1: ECD1 A,

    RetTime Sig Type    Height   Amt/Height   Amount   Grp   Name
     [min]              [counts]               [UG/ML]
    -------|--|------|----------|----------|----------|--|---------------
      6.746  1 BB  +  1.99248e5  3.01132e-7 6.00000e-2  1 SURROGATE (TCMX)
      7.833  1 VB     2.86529e4  1.74503e-5 5.00000e-1  2 1242-1
      8.521  1 VV     5.20885e4  9.59905e-6 5.00000e-1  2 1242-2
      9.528  1 VV     2.93145e4  1.70564e-5 5.00000e-1  2 1242-3
      9.692  1 VV     2.66815e4  1.87396e-5 5.00000e-1  2 1242-4
     10.050  1 VV     3.84080e4  1.30181e-5 5.00000e-1  2 1242-5
     10.909  1 VV     3.62073e4  1.38094e-5 5.00000e-1  2 1242-6
     11.455  1 VV     1.40142e4  3.56781e-5 5.00000e-1  2 1242-7
     11.674  1 VV     1.06554e4  4.69244e-5 5.00000e-1  2 1242-8
     15.212  1 BB  +  1.85437e5  3.23498e-7 5.99885e-2  1 SURROGATE (DCBP)

    Totals :                                 4.65145e5

     Results obtained with enhanced integrator!
    Group summary :

     Group  Use   Height      Amount       Group Name
      ID          [counts]    [UG/ML]
    |------|---|----------|----------|--------------------------|
      1         3.84685e5  1.19989e-1  SURROGATES
      2         2.36022e5     4.00000  AR 1242

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    ==================================================================
                          *** End of Report ***

3203A41034 3/24/2008 10:18:23 AM RBO                      Page 2 of 2
```

FIGURE 5.1

CHROMATOGRAM OF AROCLOR 1016

```
Data File D:\HPCHEM\2\DATA\GC1923\004F0401.D          Sample Name: 1016 0.5 UG/ML
    LOG 173 PAGE 186

    ==================================================================
    Injection Date  : 2/7/2008 4:04:37 PM              Seq. Line :   4
    Sample Name     : 1016 0.5 UG/ML                        Vial :   4
    Acq. Operator   : FUZ                                    Inj :   1
                                                       Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\16-1923.M
    Last changed    : 3/24/2008 11:15:50 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8081. COLUMN RESTEK RTX-35 30M X 0.35 ID X 0.50 MICRON FILM
    S/N 566535. 3ul injection with gooseneck liner.
    ==================================================================
```



3203A41034 3/24/2008 11:15:50 AM RBO Page 1 of 2

FIGURE 5.2

REPORT OF AROCLOR 1016

```
Data File D:\HPCHEM\2\DATA\GC1923\004F0401.D          Sample Name: 1016 0.5 UG/ML
    LOG 173 PAGE 186

    ==================================================================
    Injection Date  : 2/7/2008 4:04:37 PM            Seq. Line :   4
    Sample Name     : 1016 0.5 UG/ML                      Vial :   4
    Acq. Operator   : FUZ                                  Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\16-1923.M
    Last changed    : 3/24/2008 11:15:50 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8081. COLUMN RESTEK RTX-35 30M X 0.35 ID X 0.50 MICRON FILM
    S/N 566535. 3ul injection with gooseneck liner.
    ==================================================================

    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By              :      Retention Time
    Calib. Data Modified   :      Monday, March 24, 2008 11:14:17 AM
    Multiplier             :      1.0000
    Dilution               :      1.0000

    Signal 1: ECD1 A,

    RetTime Sig Type    Height    Amt/Height   Amount    Grp   Name
     [min]              [counts]               [ug/mL]
    -------|--|------|----------|----------|----------|--|---------------
      6.753  1 PB   +  1.89732e5  3.16236e-7 6.00000e-2  1 SURROGATE (TCMX)
      7.839  1 VB     3.25294e4  1.53707e-5 5.00000e-1  2 1016-1
      8.526  1 VV     6.26833e4  7.97661e-6 5.00000e-1  2 1016-2
      8.848  1 VV     2.18030e4  2.29326e-5 5.00000e-1  2 1016-3
      8.931  1 VV     2.62474e4  1.90495e-5 5.00000e-1  2 1016-4
      9.137  1 VV     1.11093e5  4.50072e-6 5.00000e-1  2 1016-5
      9.354  1 VV     4.36741e4  1.14484e-5 5.00000e-1  2 1016-6
      9.532  1 VV     3.49813e4  1.42933e-5 5.00000e-1  2 1016-7
     10.053  1 VV     4.08131e4  1.22510e-5 5.00000e-1  2 1016-8
     10.232  1 VV     2.52372e4  1.98120e-5 5.00000e-1  2 1016-9
     15.217  1 VB   +  1.61972e5  3.70434e-7 6.00000e-2  1 SURROGATE (DCBP)

    Totals :                                1.67113e5

     Results obtained with enhanced integrator!
    Group summary :

     Group  Use   Height      Amount       Group Name
      ID         [counts]    [ug/mL]
    |------|---|----------|----------|--------------------------|
      1         3.51704e5  1.20000e-1  SURROGATES
      2         3.99062e5     4.50000  AR 1016

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    ==================================================================
                            *** End of Report ***

3203A41034 3/24/2008 11:15:50 AM RBO                          Page 2 of 2
```

FIGURE 6.1

CHROMATOGRAM OF AROCLOR 1221

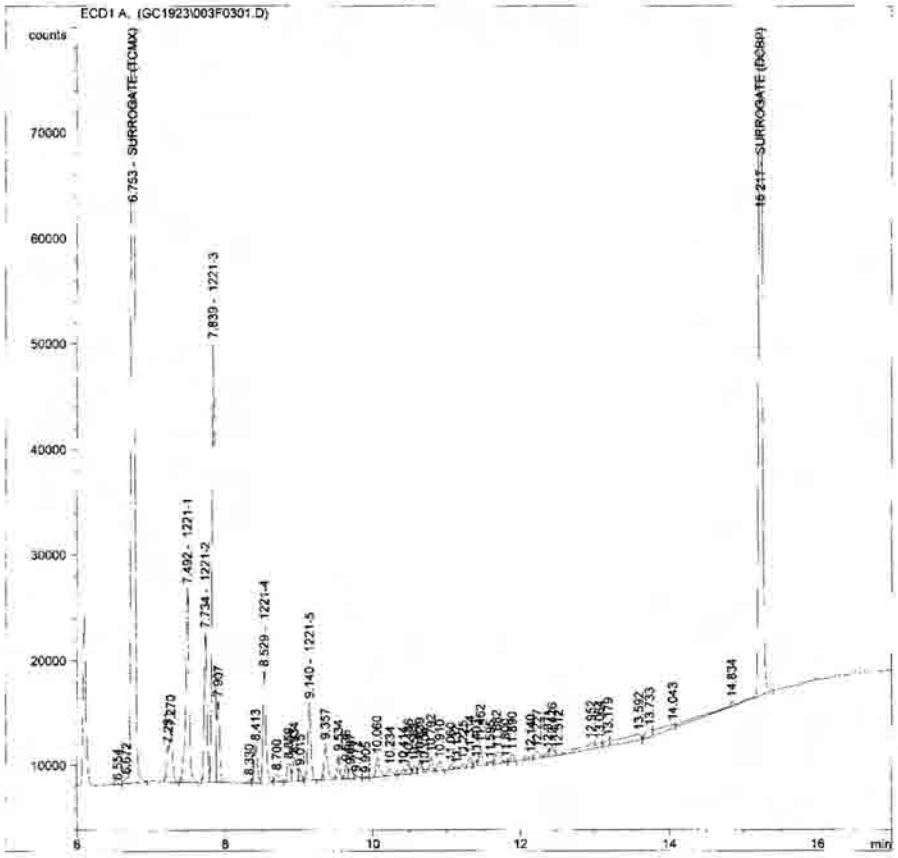
Data File D:\HPCHEM\2\DATA\GC1923\003F0301.D Sample Name: 1221 0.5 UG/ML
LOG 173 PAGE 187

==========

Injection Date	: 2/7/2008 3:39:35 PM	Seq. Line :	3
Sample Name	: 1221 0.5 UG/ML	Vial :	3
Acq. Operator	: FUZ	Inj :	1
		Inj Volume :	Manually

Acq. Method : D:\HPCHEM\2\METHODS\PCB.M
Last changed : 2/7/2008 2:46:21 PM by FUZ
Analysis Method : D:\HPCHEM\2\METHODS\21-0917.M
Last changed : 3/24/2008 11:07:47 AM by RBO
(modified after loading)
FIELD PCB'S BY 8081. COLUMN RESTEK RTX-35 30M X 0.35 ID X 0.50 MICRON FILM S/N 566535. 3ul injection with gooseneck liner.

==========



3203A41034 3/24/2008 11:07:47 AM RBO Page 1 of 2

FIGURE 6.2

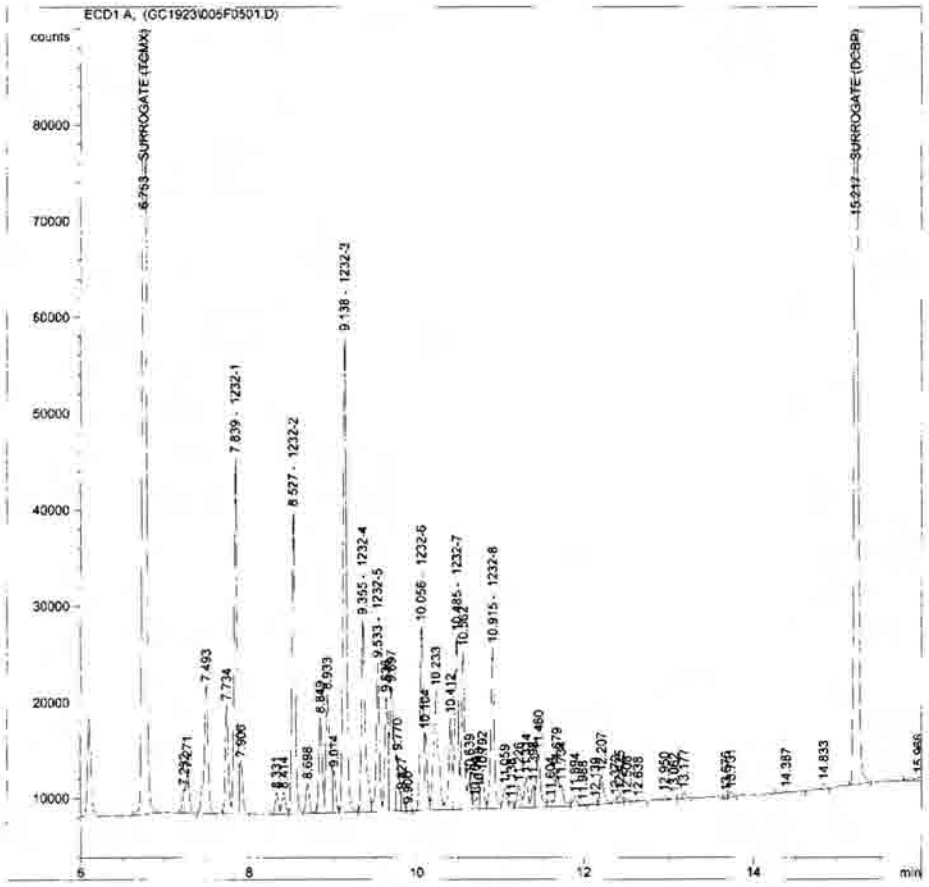
REPORT OF AROCLOR 1221

```
Data File D:\HPCHEM\2\DATA\GC1923\003F0301.D          Sample Name: 1221 0.5 UG/ML
    LOG 173 PAGE 187

    ==================================================================
    Injection Date  : 2/7/2008 3:39:35 PM            Seq. Line :   3
    Sample Name     : 1221 0.5 UG/ML                      Vial :   3
    Acq. Operator   : FUZ                                  Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\21-0917.M
    Last changed    : 3/24/2008 11:07:44 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8081. COLUMN RESTEK RTX-35 30M X 0.35 ID X 0.50 MICRON FILM
    S/N 566535. 3ul injection with gooseneck liner.
    ==================================================================

    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By              :      Retention Time
    Calib. Data Modified   :      Monday, March 24, 2008 11:05:19 AM
    Multiplier             :      1.0000
    Dilution               :      1.0000

    Signal 1: ECD1 A,

    RetTime Sig Type    Height   Amt/Height   Amount   Grp   Name
     [min]             [counts]               [ug/mL]
    -------|--|------|----------|----------|----------|--|---------------
      6.753  1 VB  +  1.86663e5  3.21435e-7 6.00000e-2  1 SURROGATE (TCMX)
      7.492  1 BB     1.85375e4  2.69723e-5 5.00000e-1  2 1221-1
      7.734  1 BV     1.43707e4  3.47930e-5 5.00000e-1  2 1221-2
      7.839  1 VV     4.17921e4  1.19640e-5 5.00000e-1  2 1221-3
      8.529  1 VB     1.07189e4  4.66467e-5 5.00000e-1  2 1221-4
      9.140  1 VB     7491.31885 6.67439e-5 5.00000e-1  2 1221-5
     15.217  1 VB  +  1.58031e5  3.79673e-7 6.00000e-2  1 SURROGATE (DCBP)

    Totals :                                 5.87054e4

     Results obtained with enhanced integrator!
    Group summary :

     Group  Use   Height      Amount       Group Name
      ID         [counts]     [ug/mL]
    |------|---|----------|----------|--------------------------|
      1         3.44694e5  1.20000e-1  SURROGATES
      2         9.29106e4     2.50000  AR 1221

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    ==================================================================
                          *** End of Report ***

3203A41034 3/24/2008 11:07:44 AM RBO                    Page 2 of 2
```

FIGURE 7.1

CHROMATOGRAM OF AROCLOR 1232

```
Data File D:\HPCHEM\2\DATA\GC1923\005F0501.D          Sample Name: 1232 0.5 UG/ML
    LOG 173 PAGE 188

    ==================================================================
    Injection Date  : 2/7/2008 4:29:34 PM              Seq. Line :   5
    Sample Name     : 1232 0.5 UG/ML                        Vial :   5
    Acq. Operator   : FUZ                                    Inj :   1
                                                       Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\32-1923.M
    Last changed    : 3/24/2008 11:22:29 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8081. COLUMN RESTEK RTX-35 30M X 0.35 ID X 0.50 MICRON FILM
    S/N 566535. 3ul injection with gooseneck liner.
    ==================================================================
```



3203A41034 3/24/2008 11:22:29 AM RBO Page 1 of 2

FIGURE 7.2

REPORT OF AROCLOR 1232

```
Data File D:\HPCHEM\2\DATA\GC1923\005F0501.D          Sample Name: 1232 0.5 UG/ML
    LOG 173 PAGE 188

    ==================================================================
    Injection Date  : 2/7/2008 4:29:34 PM            Seq. Line :   5
    Sample Name     : 1232 0.5 UG/ML                      Vial :   5
    Acq. Operator   : FUZ                                  Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\32-1923.M
    Last changed    : 3/24/2008 11:22:29 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8081. COLUMN RESTEK RTX-35 30M X 0.35 ID X 0.50 MICRON FILM
    S/N 566535. 3ul injection with gooseneck liner.
    ==================================================================

    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By              :      Retention Time
    Calib. Data Modified   :      Monday, March 24, 2008 11:21:02 AM
    Multiplier             :      1.0000
    Dilution               :      1.0000
    Sample Amount          :      1.00000  [UG/ML]   (not used in calc.)

    Signal 1: ECD1 A,

    RetTime Sig Type    Height    Amt/Height   Amount    Grp   Name
     [min]             [counts]                [UG/ML]
    -------|--|------|----------|----------|----------|--|---------------
      6.753  1 PB  +  1.91562e5  3.13215e-7 6.00000e-2  1 SURROGATE (TCMX)
      7.839  1 VV     3.71447e4  1.34609e-5 5.00000e-1  2 1232-1
      8.527  1 VV     3.15942e4  1.58257e-5 5.00000e-1  2 1232-2
      9.138  1 VB     4.96083e4  1.00790e-5 5.00000e-1  2 1232-3
      9.355  1 BV     2.01164e4  2.48553e-5 5.00000e-1  2 1232-4
      9.533  1 VV     1.56576e4  3.19334e-5 5.00000e-1  2 1232-5
     10.056  1 VV     1.93120e4  2.58907e-5 5.00000e-1  2 1232-6
     10.485  1 VV     1.81785e4  2.75050e-5 5.00000e-1  2 1232-7
     10.915  1 VV     1.68438e4  2.96845e-5 5.00000e-1  2 1232-8
     15.217  1 BB  +  1.65727e5  3.62042e-7 6.00000e-2  1 SURROGATE (DCBP)

    Totals :                                 1.94381e5

     Results obtained with enhanced integrator!
    Group summary :

     Group  Use   Height      Amount       Group Name
      ID         [counts]     [UG/ML]
    |------|---|----------|----------|--------------------------|
      1          3.57288e5  1.20000e-1  SURROGATES
      2          2.08455e5     4.00000  AR 1242

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    ==================================================================
                            *** End of Report ***

3203A41034 3/24/2008 11:22:29 AM RBO                          Page 2 of 2
```

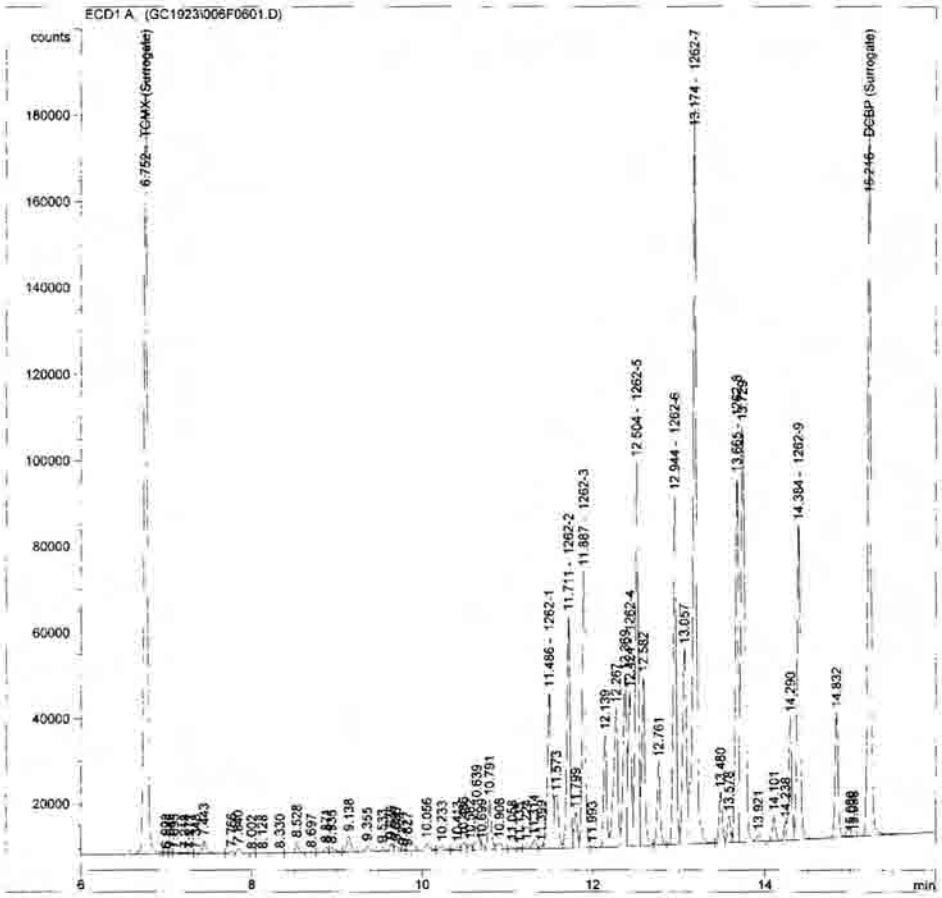
FIGURE 8.1

CHROMATOGRAM OF AROCLOR 1262

Data File D:\HPCHEM\2\DATA\GC1923\006F0601.D Sample Name: 1262 0.5 UG/ML
LOG 173 PAGE 189

Injection Date	: 2/7/2008 4:55:26 PM	Seq. Line :	6
Sample Name	: 1262 0.5 UG/ML	Vial :	6
Acq. Operator	: FUZ	Inj :	1
		Inj Volume :	Manually
Acq. Method	: D:\HPCHEM\2\METHODS\PCB.M		
Last changed	: 2/7/2008 2:46:21 PM by FUZ		
Analysis Method	: D:\HPCHEM\2\METHODS\62-1923.M		
Last changed	: 3/24/2008 10:54:51 AM by RBO (modified after loading)		

FIELD PCB'S BY 8082. COLUMN RESTEK RTX-35 30M X 0.53 ID X 0.5 MICRON FILN S/N (482893). 2uL injection



3203A41034 3/24/2008 10:54:52 AM RBO Page 1 of 2

FIGURE 8.2

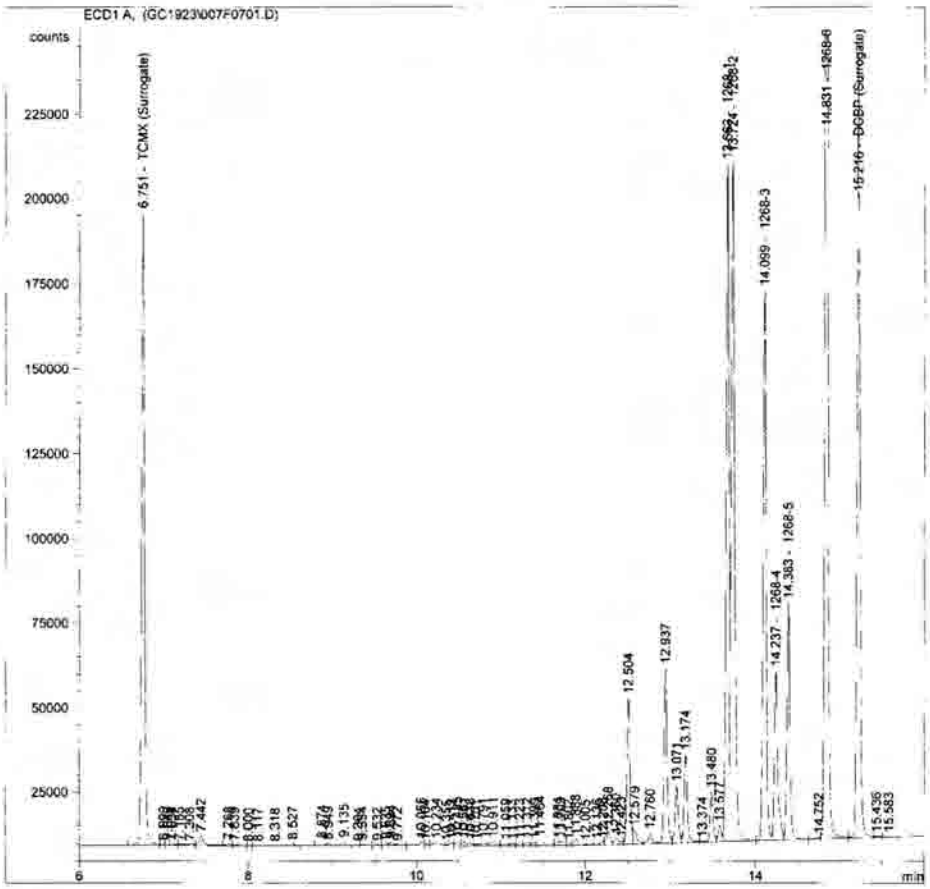
REPORT OF AROCLOR 1262

```
Data File D:\HPCHEM\2\DATA\GC1923\006F0601.D          Sample Name: 1262 0.5 UG/ML
    LOG 173 PAGE 189

    ==================================================================
    Injection Date  : 2/7/2008 4:55:26 PM            Seq. Line :   6
    Sample Name     : 1262 0.5 UG/ML                      Vial :   6
    Acq. Operator   : FUZ                                  Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\62-1923.M
    Last changed    : 3/24/2008 10:54:51 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8082. COLUMN RESTEK RTX-35 30M X 0.53 ID X 0.5 MICRON FILN S/
    N (482893). 2uL  injection
    ==================================================================

    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By              :      Retention Time
    Calib. Data Modified   :      3/21/2008 10:00:12 AM
    Multiplier             :      1.0000
    Dilution               :      1.0000
    Sample Amount          :      1.00000  [UG/ML]   (not used in calc.)

    Signal 1: ECD1 A,

    RetTime Sig Type    Height    Amt/Height   Amount   Grp   Name
     [min]              [counts]               [UG/ML]
    -------|--|------|----------|----------|----------|--|---------------
      6.752  1 BV     1.92462e5  3.11750e-7 6.00000e-2  1 TCMX (Surrogate)
     11.486  1 VV     3.63862e4  1.37415e-5 5.00000e-1  2 1262-1
     11.711  1 VV     5.40776e4  9.24598e-6 5.00000e-1  2 1262-2
     11.887  1 VV     6.43538e4  7.76954e-6 5.00000e-1  2 1262-3
     12.424  1 VV     3.58817e4  1.39347e-5 5.00000e-1  2 1262-4
     12.504  1 VV     8.94137e4  5.59198e-6 5.00000e-1  2 1262-5
     12.944  1 VV     8.13914e4  6.14316e-6 5.00000e-1  2 1262-6
     13.174  1 VV     1.73569e5  2.88069e-6 5.00000e-1  2 1262-7
     13.665  1 VV     8.49494e4  5.88586e-6 5.00000e-1  2 1262-8
     14.384  1 VV     7.32312e4  6.82769e-6 5.00000e-1  2 1262-9
     15.216  1 VV  +  1.69059e5  3.54906e-7 6.00000e-2  1 DCBP (Surrogate)

    Totals :                                 4.64641e5

     Results obtained with standard integrator!
    Group summary :

     Group  Use   Height      Amount       Group Name
      ID         [counts]    [UG/ML]
    |------|---|----------|----------|--------------------------|
      1         3.61521e5  1.20000e-1  Surrogate
      2         6.93254e5     4.50000  Aroclor-1260

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    ==================================================================
                            *** End of Report ***

3203A41034 3/24/2008 10:54:52 AM RBO                          Page 2 of 2
```

FIGURE 9.1

CHROMATOGRAM OF AROCLOR 1268

```
Data File D:\HPCHEM\2\DATA\GC1923\007F0701.D          Sample Name: 1268 0.5 UG/ML
   LOG 173 PAGE 189

   ==================================================================
   Injection Date  : 2/7/2008 5:20:20 PM              Seq. Line :   7
   Sample Name     : 1268 0.5 UG/ML                        Vial :   7
   Acq. Operator   : FUZ                                    Inj :   1
                                                     Inj Volume : Manually
   Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
   Last changed    : 2/7/2008 2:46:21 PM by FUZ
   Analysis Method : D:\HPCHEM\2\METHODS\68-1923.M
   Last changed    : 3/24/2008 10:58:38 AM by RBO
                     (modified after loading)
   FIELD PCB'S BY 8082. COLUMN RESTEK RTX-35 30M X 0.53 ID X 0.5 MICRON FILN S/
   N (482893). 2uL  injection
   ==================================================================
```



3203A41034 3/24/2008 10:58:38 AM RBO Page 1 of 2

FIGURE 9.2

REPORT OF AROCLOR 1268

```
Data File D:\HPCHEM\2\DATA\GC1923\007F0701.D          Sample Name: 1268 0.5 UG/ML
    LOG 173 PAGE 189

    ==================================================================
    Injection Date  : 2/7/2008 5:20:20 PM            Seq. Line :   7
    Sample Name     : 1268 0.5 UG/ML                      Vial :   7
    Acq. Operator   : FUZ                                  Inj :   1
                                                     Inj Volume : Manually
    Acq. Method     : D:\HPCHEM\2\METHODS\PCB.M
    Last changed    : 2/7/2008 2:46:21 PM by FUZ
    Analysis Method : D:\HPCHEM\2\METHODS\68-1923.M
    Last changed    : 3/24/2008 10:58:38 AM by RBO
                      (modified after loading)
    FIELD PCB'S BY 8082. COLUMN RESTEK RTX-35 30M X 0.53 ID X 0.5 MICRON FILN S/
    N (482893). 2uL  injection
    ==================================================================

    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By              :      Retention Time
    Calib. Data Modified   :      3/21/2008 10:18:05 AM
    Multiplier             :      1.0000
    Dilution               :      1.0000
    Sample Amount          :      1.00000  [UG/ML]   (not used in calc.)

    Signal 1: ECD1 A,

    RetTime Sig Type    Height    Amt/Height   Amount   Grp   Name
     [min]             [counts]                [UG/ML]
    -------|--|------|----------|----------|----------|--|---------------
      6.751  1 BV     1.86554e5  3.21623e-7 6.00000e-2  1 TCMX (Surrogate)
     13.663  1 VV     1.99964e5  2.50045e-6 5.00000e-1  2 1268-1
     13.724  1 VV     2.01753e5  2.47828e-6 5.00000e-1  2 1268-2
     14.099  1 VV     1.62467e5  3.07754e-6 5.00000e-1  2 1268-3
     14.237  1 VV     5.00465e4  9.99071e-6 5.00000e-1  2 1268-4
     14.383  1 VV     6.99480e4  7.14816e-6 5.00000e-1  2 1268-5
     14.831  1 VV     4.39721e5  1.13708e-6 5.00000e-1  2 1268-6
     15.216  1 VV  +  2.31672e5  2.58986e-7 6.00000e-2  1 DCBP (Surrogate)

    Totals :                                2.06478e5

     Results obtained with standard integrator!
    Group summary :

     Group  Use   Height      Amount       Group Name
      ID         [counts]    [UG/ML]
    |------|---|----------|----------|--------------------------|
      1         4.18226e5  1.20000e-1  Surrogate
      2         1.12390e6     3.00000  Aroclor-1260

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    ==================================================================
                            *** End of Report ***

3203A41034 3/24/2008 10:58:38 AM RBO                          Page 2 of 2
```

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

Reviewed By:

Richard W Johnson

07/07/11

Richard Johnson, Senior Chemist — Date

Michael Linskens

07/07/11

Michael Linskens, Quality Manager — Date

Approved By:

07/07/11

Nick Nigro, President — Date

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Copy No. uncontrolled copy

TESTAMERICA KNOXVILLE
SOP CHANGE FORM

SOP NUMBER: KNOX-ID-0013, Rev ~~10~~ 11 VMK 5/7/12

SOP TITLE: Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS

SOP SECTIONS AFFECTED BY CHANGE: Section 11.3.1

REASON FOR ADDITION OR CHANGE: To correct MID start time from 8 to 11 minutes

CHANGE EFFECTIVE FROM: 5/7/12

CHANGE OR ADDITION (SPECIFY SECTION; USE ADDITIONAL SHEETS IF NECESSARY)

Section 11.3.1: Change MID start time form 8 to 11 minutes

SUBMITTED BY/DATE: Daniel Gabriel 5-7-12

APPROVED BY:

Technical Reviewer Signature	Date 5-7-12
Environmental Health & Safety Signature	Date 5-8-12
QA Signature	Date 5-8-112
Management Signature	Date 5/8/12

TESTAMERICA KNOXVILLE

STANDARD OPERATING PROCEDURE

TITLE: Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS

(SUPERSEDES: KNOX-ID-0013, Revision 10)

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1 Scope and Application

1.1 This procedure is designed to meet analytical program requirements where HRGC/HRMS analysis of polychlorinated biphenyl (PCB) isomers is specified. The procedure is used by TestAmerica Knoxville for the qualitative and quantitative measurement of all 209 PCB congeners in a variety of environmental matrices at part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations. This procedure is based on EPA method 1668A, 1668B and 1668C.

1.2 The compounds listed in Table 1 may be determined by this procedure. The detection limits and quantitation levels in this method are usually dependent on the level of interferences rather than instrumental limitations. The estimated minimum levels (EMLs) in Table 4 are the levels at which the PCBs can be determined with only common laboratory interferences present. The actual limits of detection and quantitation will vary depending on the complexity of the matrix.

1.3 The Low Calibration Levels (LCLs) of the method are listed in Table 3 for individual congeners. Analysis of a one-tenth aliquot of the sample permits measurements of concentrations up to 10 times the upper calibration range. Samples containing concentrations of PCBs that are greater than ten times the upper calibration are analyzed by protocols designed for such concentration levels.

1.4 The GC/MS portions of this method are for use only by analysts experienced with HRGC/HRMS or under the close supervision of such qualified persons. Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in section 9.1.

1.5 This procedure is a "performance-based" method. These reference methods allow modifications to overcome interferences or lower the cost of measurements, if all performance criteria in the methods are met and method equivalency is established. Deviations from the referenced methods have been incorporated into this procedure and are listed in section 17.1. Deviations to this procedure are only allowed as specified in section 11.1.

1.6 Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCBs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed. Section 5 of this procedure discusses safety procedures.

2 Summary of Method

2.1 All solid, semi-solid and tissue samples are screened by GC/ECD prior to extraction. Aqueous samples may be screened if the potential for congener levels above 40 ng/L exists. Variations in sample size, spiking levels and final volume are established based on the screening result.

2.2 After sample extraction, cleanup, and concentration, recovery standards are added to each extract, and an aliquot of the extract is injected into the gas chromatograph. The analytes are separated by the GC and detected by a high-resolution (≥10,000) mass spectrometer. Two exact masses are monitored for each analyte.

2.3 An individual PCB congener is identified by comparing the GC retention time and ion-abundance ratio of two exact masses with the corresponding retention time of an authentic standard and the theoretical or acquired ion-abundance ratio of the two exact masses.

2.4 Quantitative analysis is performed using selected ion current profile (SICP) areas using the internal standard technique.

2.5 The quality of the analysis is assured through reproducible calibration and verification of the extraction, cleanup, and GC/MS systems.

3 Definitions

These definitions and purposes are specific to this method but conform to common usage as much as possible.

Note: Terminology differences existing in some isotope dilution reference methods regarding the functionality of the labeled analogs may lead to confusion. For example, EPA's Office of Solid Waste methods (8280, 8290) use the term "Internal Standards" to describe the labeled analogs which are added to the sample prior to extraction and used to quantitate the native targets. EPA's Office of Water methods (1613B, 1668) use the term "Labeled Analogs" to describe these same compounds while using the term "Internal Standards" to describe the labeled analogs which are added to the extract just prior to analysis and used to quantitate the recovery of the labeled analogs added before extraction. EPA's Office of Solid Waste methods (8280, 8290) uses the term "Recovery Standards" to describe these later labeled analogs.

The terminology conventions established by the EPA's Office of Solid Waste methods (8280, 8290) are used in the laboratory for all Standard Operating Procedures and internal communications as defined in this section.Analyte – A PCB tested for by this method. The analytes are listed in Table 1.

3.1 Calibration verification standard (VER) – The mid-point calibration standard (CS3) that is used to verify calibration. See Table 6a.

3.2 CB – Chlorinated biphenyl congener. One of the 209 individual chlorinated biphenyl congeners determined using this method. The 209 CBs are listed in Table 1.

3.3 Cleanup Standard – Isotopically labeled compounds that are added to samples, blanks, quality control samples, and calibration solutions. They are added to the samples after extraction but prior to extract cleanup, and are used to assess the efficiency of the cleanup procedures.

3.4 Congener – Any member of a particular homologous series, for example, 2,2’-DiCB.

3.5 CS0.5, CS1, CS2, CS3, CS4, CS5 – See Calibration standards and Table 6a.

3.6 Estimated Detection Limit (EDL) – The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level (noise level).

3.7 Estimated Maximum Possible Concentration (EMPC) – The calculated concentration of a signal having the same retention time as a PCB congener but which does not meet the other qualitative identification criteria defined in the method.

3.8 Estimated Minimum Level (EML) – The lowest concentration at which an analyte can be measured reliably with common laboratory interferences present. This is the reporting limit (RL)

3.9 Field blank – An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratory or the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.

3.10 GC – Gas chromatograph or gas chromatography.

3.11 Homologous Series – A series of compounds in which each member contains the same number of chlorine atoms. The members of the series are called homologs.

3.12 HRGC – High resolution GC.

3.13 HRMS – High resolution MS.

3.14 ICV – Initial Calibration Verification Standard. A calibration standard from a second source, traceable to a national standard if possible. The ICV is analyzed after the Initial calibration to verify the concentration of the initial calibration standards.

3.15 Internal Standards (IS) – Isotopically labeled analogs of the target analytes that are added to every sample, blank, quality control spike sample, and calibration solution. They are added to the sample before extraction and are used to calculate the concentration of the target analytes or detection limits.

3.16 IPR (also known as IDOC)– Initial precision and recovery; four aliquots of the PAR standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed prior to the first time this method is used and any time the method or instrumentation undergoes significant modification.

3.17 Isomer – PCB congeners that contain the same number of chlorine atoms, but differ in the structural arrangement of the chlorine atoms. For example, PCB-4 and PCB-9 are isomers.

3.18 Laboratory blank – See Method blank.

3.19 Laboratory control sample (LCS) – See ongoing precision and recovery standard (OPR).

3.20 Laboratory reagent blank – See method blank.

3.21 Level of Chlorination (LOC) Congeners – The first and last eluting congeners in each homolog (or level of chlorination). (For the SPB-Octyl Column the LOC Congeners are 1, 3; 4, 15; 19, 37; 54, 77; 104, 126; 155, 169; 188, 189; 202, 205; 208, 206; 209)

3.22 Method blank – An aliquot of a clean test matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

3.23 Minimum Level (ML) – The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

3.24 Note: EML is the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present

3.25 MS – Mass spectrometer or mass spectrometry.

3.26 OPR (also know as ODOC) – Ongoing precision and recovery standard (OPR); a laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

3.27 PAR – Precision and recovery standard; secondary standard that is diluted and spiked to form the IPR and OPR.

3.28 PFK – Perfluorokerosene; the mixture of compounds used to calibrate the exact mass scale in the HRMS.

3.29 Primary dilution standard – A solution containing the specified analytes that is purchased or prepared from stock solutions and diluted as needed to prepare calibration solutions and other solutions.

3.30 Quality control check sample (QCS) – A sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

3.31 PCB – Polychlorinated biphenyl.

3.32 Reagent water – Water demonstrated to be free from the analytes of interest and potentially interfering substances at the method minimum level for the analyte.

3.33 Recovery Standard (RS) – Isotopically labeled compounds which are added to every sample, blank, and quality control spike sample extract prior to analysis. They are used to measure the recovery of the internal standards and the cleanup standards.

3.34 Relative Percent Difference (RPD) – A measure of the difference between two values normalized to one of the values. It is used to determine the accuracy of the concentration measurements of second source verification standards.

3.35 Relative standard deviation (RSD) – The standard deviation times 100 divided by the mean. Also termed "coefficient of variation."

3.36 RF or RRF (Relative Response Factor) - The ratio of the response of the mass spectrometer to a known amount of a compound relative to that of a known amount of a reference standard as measured in the initial and continuing calibrations. It is used to determine instrument

performance and it is used to calculate the concentration of target analytes, internal standard recoveries, or detection limits in samples, blanks, and quality control samples. See Section 10.3.4.2.

3.37 SICP – Selected ion current profile.

3.38 SPE – Solid-phase extraction; an extraction technique in which an analyte is extracted from an aqueous sample by passage over or through a material capable of reversibly adsorbing the analyte. Also termed liquid-solid extraction.

3.39 Specificity – The ability to measure an analyte of interest in the presence of interferences and other analytes of interest encountered in a sample.

3.40 Stock solution - A solution containing an analyte that is prepared using a reference material traceable to EPA, the National Institute of Science and Technology (NIST), or a source that will attest to the purity and authenticity of the reference material.

3.41 Surrogate Standards (SS) - Isotopically labeled compounds that are added to XAD samples and calibration solution. They are added to XAD sampling tubes before sampling and are used to measure sampling and recovery efficiency.

3.42 Toxic Congeners (or Toxic Isomers) – PCBs determined by the World Health Organization and USEPA to have dioxin-like toxicity. (PCBs 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, 189)

3.43 Toxic/LOC Congeners – PCBs belonging to either the Toxic Congeners list or the LOC Congeners list.

3.44 VER – See Calibration verification standard.

3.45 Additional definitions can be found in the TestAmerica Knoxville Quality Assurance Manual (QAM), current revision.

4 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. Where possible, glassware is cleaned by extraction or solvent rinse. The non-coplanar PCB congeners 105, 114, 118, 123, 156, 157, 167, and 180 have been shown to be very difficult to completely eliminate from the laboratory at the minimum levels in this method, and baking of glassware in a kiln or furnace at 450 - 500°C may be necessary to remove these and other contaminants.

4.2 All materials used in the analysis shall be demonstrated to be free from interferences by running laboratory method blanks (section 9.5) initially and with each sample batch.

4.3 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxy-diphenyl ethers, benzylphenyl ethers, polynuclear aromatics, and pesticides. Because

very low levels of PCBs are measured by this method, the elimination of interferences is essential. Cleanup steps can be used to reduce or eliminate these interferences and thereby permit reliable determination of the PCBs at the levels shown in Table 3.

5 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Corporate Safety Manual), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.

5.3 The effluents of sample splitters for the gas chromatograph and roughing pumps on the mass spectrometer must be vented to the laboratory hood exhaust system or must pass through an activated charcoal filter.

5.4 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them or use thermal protection when working on them while they are above room temperature.

5.5 The mass spectrometer is under high vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. Alternatively, the source may be removed from the vacuum manifold through a vacuum interlock.

5.6 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power. If the work involved requires measurement of voltage supplies, the instrument may be left on.

5.7 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen, Irritant	25 ppm-TWA, 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hexane	Flammable, Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable, Poison, Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Toluene	Flammable, Poison, Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Nonane	Flammable	None established	Harmful if inhaled/swallowed. Vapor/mist is irritating to eyes, mucous memebranes and upper respiratory tract. Causes skin irritiation.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.8 Chemicals that have been classified as **carcinogens**, **potential carcinogens**, or **mutagens include**: methylene chloride, polychlorinated biphenyls, and toluene. The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.)

5.9 Chemicals known to be **flammable** are: acetone, hexane, nonane and toluene.

5.10 Exposure to chemicals will be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.11 The preparation of all standards will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.12 Personal Hygiene: Thorough washing of hands and forearms is recommended after each manipulation and before breaks (coffee, lunch, and shifts).

5.13 Confinement: Work areas should be isolated and posted with signs. Glassware and tools should be segregated. Bench tops should be covered with plastic backed absorbent paper.

5.14 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.

5.15 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

5.16 All work must be stopped in the event of a known or potential compromise to the health or safety of laboratory personnel. The situation must be reported immediately to a laboratory supervisor.

6 Equipment and Supplies

6.1 Gas chromatograph - Shall have splitless or on-column injection port for capillary column, temperature program with isothermal hold, and shall meet all of the performance specifications in Section 10.

6.1.1 Column #1 – 30±5-m long x 0.25±0.02-mm ID; 0.25-µm film SPB-Octyl (Supelco 2-4218, or equivalent).

6.2 Mass spectrometer - Electron impact ionization, shall be capable of repetitively selectively monitoring 20 exact masses minimum at high resolution (≥10,000) during a period less than 1.0 second, and shall meet all of the performance specifications in Section 10.

6.3 GC/MS interface - The mass spectrometer (MS) shall be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beams.

6.4 Data system - Capable of collecting, recording, and storing MS data.

7 Reagents and Standards

CAUTION: Refer to Material Safety Data Sheets (MSDS) for specific safety information on chemicals and reagents prior to use or as needed.

CAUTION: During preparation of reagents, associates shall wear lab coat, gloves, safety glasses with side shields, and laboratory approved shoes as a minimum. Reagents shall be prepared in a fume hood.

7.1 Solvents - Acetone, toluene, n-hexane, methanol, methylene chloride, and nonane; pesticide quality.

7.2 Perfluorokerosene (PFK) high boiling mass spectroscopy grade; bp 210-260°C; d^{20}_4 1.94; n^{20}_D 1.330; Fluka (Catalog No. - 77275).

7.3 $^{13}C_{12}$ Labeled PCB Congener Standards: Obtained as individual Certified Reference Standards from Cambridge Isotope Laboratories (CIL, Andover Massachusetts) and Wellington Laboratories (Guelph, Ontario, Canada). (Refer to Table 5b for a list of individual standards.) These standards are purchased at 40 µg/mL or 50 µg/mL in nonane. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a Teflon®-lined screw cap. When not being used, standards are stored in a dark box at room temperature. These purchased standards are used to prepare the following mixed stock solutions and spiking solutions:

7.3.1 Internal Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled internal standards listed in Table 5b, to a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.3.2 Internal Standard Spiking Solution: Prepared by diluting the 1000 ng/mL internal standard stock solution to a concentration of 10 ng/mL in acetone. One to 4.0 mL of this solution is added to each solid/tissue sample prior to extraction. Refer to Table 12, “Assignment of Sample Preparation Protocols” to determine the exact volume to add. Twenty uL of the internal standard spiking solution is added to each aqueous sample prior to extraction.

7.3.3 Recovery Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled recovery standards listed in Table 5b to a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.3.4 Recovery Standard Spiking Solution: Prepared by diluting the 1000 ng/mL recovery standard stock solution to a concentration of 100 ng/mL in nonane. Fifty to 100 µL of this spiking solution is added to each solid/tissue sample extract prior to analysis (refer to Table 12), whereas, 20 µL is added to each aqueous sample extract.

7.3.5 Cleanup Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled cleanup standards listed in Table 5b, to a concentration of 5000 ng/mL in

nonane. The concentration is verified by GC/MS before use.

7.3.6 Cleanup Standard Spiking Solution: Prepared by diluting the 5000 ng/mL cleanup standard stock solution to a concentration of 10 ng/mL in hexane. One half to 1.0 mL of this solution is added to each solid/tissue sample extract prior to cleanup (refer to Table 12), whereas, 0.20 mL is added to each aqueous sample extract.

7.3.7 Sampling Surrogate Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled sampling surrogate standards listed in Table 5b to a concentration of 5000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.3.8 Sampling Surrogate Spiking Solution: Prepared by diluting the 5000 ng/mL sampling surrogate stock solution to a concentration of 50 ng/mL in nonane.

7.4 Native PCB Congener Standard Mix: Obtained as a Certified Reference Standard from Accustandard (New Haven, CT). This standard contains all 209 PCB congeners at 4000 ng/mL in nonane. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a Teflon®-lined screw cap. When not being used, the standard is stored in a dark box at room temperature. This purchased standard is used to prepare the following native stock solution and spiking solution:

7.4.1 Native PCB Congener Stock Solution: Prepared by diluting the 4000 ng/mL native PCB congener standard mix to a concentration of 40 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.4.2 LCS Spiking Solution: Prepared by diluting the 4000 ng/mL native PCB congener standard to a concentration of 5.0 ng/mL in acetone. One mL of this solution is added to each solid/tissue LCS prior to extraction, whereas, 0.20 mL is added to each aqueous LCS.

7.5 Calibration Standard Solutions (CS 0.5 through CS 5) are prepared by dilution of the native PCB congener standards in section 7.4 and 7.4.1 and the labeled standards in section 7.3 in nonane. Table 6a shows the calibration solution analytes and final concentrations. Table 6b provides details for preparation of these calibration solutions.

7.5.1 This series of solutions is used to establish linearity and relative response factors for all compounds in the initial calibration solutions. These RRFs are used to quantify PCB congeners in the calibration verification (VER) and all samples. The CS3 standard is used for calibration verification. The VER solution is also used to verify chromatographic performance.

7.6 PCB Congener Mix 1 through 5 standard solutions containing all 209 isomers are Certified Reference Standards (Accustandard Product No's. M-1668A-1, M-1668A-2, M-1668A-3, M-1668A-4, M-1668A-5). Stock solutions are purchased at 250-750 µg/mL in isooctane. Once the ampoule has been sonicated and opened, the solution is transferred to an amber glass vial with Teflon®-lined cap and is used as received. These five mixes are run in triplicate to determine the retention times for each of the congeners and which congeners will co-elute for each new SPB Octyl column used.

7.6.1 209 PCB ICAL Verification stock solution: Prepared by combining the five PCB Congener Mixes referred to in section 7.6 and diluting to a concentration of 5000-15000 ng/mL in nonane.

7.6.2 Initial Calibration Verification Standard. This is a single solution containing all 209 individual PCBs as well as internal standards and recovery standards at the following concentrations:

- mono, di, and tri CBs at 50 ng/mL
- tetra, penta, hexa and hepta CBs at 100 ng/mL
- octa, nona, and deca CBs at 150 ng/mL
- internal standards and recovery standards are at the same concentration as the calibration standards (CS 0.5 – CS 5).

This solution is always analyzed immediately after the initial calibration.

7.6.2.1 Combine 100 uL of 209 PCB ICAL verification stock solution (Section 7.6.1) (equivalent to 20 uL of each mix) with 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled internal standard stock solution, 100 uL of a 1/10 dilution of the 5000 ng/mL $^{13}C_{12}$ labeled cleanup standard stock solution, 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled recovery standard stock solution and 600 uL of nonane to produce the concentrations listed in section 7.6.2.

7.6.3 Retention Time Calibration Mixes: These are 5 solutions injected in triplicate to establish the retention time data referenced in section 10.2.3. Combine 20 uL of the Accustandard PCB Congener Mix 1 (Section 7.6) with 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled internal standard stock solution, 100 uL of a 1/10 dilution of the 5000 ng/mL $^{13}C_{12}$ labeled cleanup standard stock solution, 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled recovery standard stock solution and 600 uL of nonane to produce the concentrations listed in section 7.6.2. Repeat the process using the Accustandard PCB Congener Mixes 2 through 5.

7.7 QC Check Sample - A QC Check Sample should be obtained from a source independent of the calibration standards. This check sample is a certified standard reference material (SRM) containing the PCBs in known concentrations in a sample matrix similar to the matrix under test. The National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland has an SRM 1944 – New York/New Jersey Waterway Sediment that the NYSDEC recommends for use.

8 Sample Collection, Preservation and Storage

8.1 Sampling is not performed for this method by TestAmerica Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, “Sample Receipt and Login”, current revision.

8.2 Holding Times

8.2.1 Store sample extracts in a dark box at room temperature until analyzed. If stored in the dark at room temperature, sample extracts may be stored for up to one year.

9 Quality Control

9.1 Initial precision and recovery (IPR) or initial demonstration of capabilty (IDOC) samples are analyzed to demonstrate the ability to generate acceptable precision and accuracy.

9.1.1 For aqueous samples, extract, clean, concentrate, and analyze four 1-L aliquots of reagent water spiked with internal standards, cleanup standard, recovery standard and the LCS spiking solution, according to the procedures in section 11. For solid/tissue samples, extract, clean, concentrate, and analyze four aliquots of sodium sulfate/corn oil spiked with internal standards, cleanup standard, recovery standard and the LCS spiking solution, according to the procedures in section 11. All steps that are to be used for processing samples, including preparation, extraction and cleanup, shall be included in this test.

9.1.2 Using the results of the set of four analyses, compute the average percent recovery (%R) of the extracts and the relative standard deviation (%RSD) of the concentration in ng/L (aqueous) and ng/g (solid) for each compound.

9.1.3 For each PCB and labeled compound, compare the %RSD and %R with the corresponding limits for initial precision and recovery in the approprirate Table 10A or 10B. If the RSD and %R for all compounds meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, any individual %RSD exceeds the precision limit or any individual %R falls outside the range for accuracy, system performance is unacceptable for that compound. Correct the problem and repeat the test.

9.2 Internal Standards

9.2.1 Every sample, blank, and QC sample is spiked with internal standards. Internal standard recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. When properly applied, results from isotope dilution techniques are independent of recovery. The recovery of each internal standard should be within the limits in Table 10A or 10B depending on the method revision being used. If the recovery is outside these limits the following corrective action should be taken:

- Check all calculations for error.
- Ensure that instrument performance is acceptable; if applicable, a daily CCV will be analyzed to confirm instrument performance.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- If the recovery of any internal standard is less than the limits indicated in Tables 10A and 10B, calculate the S/N ratio of the internal standard. If the S/N is > 10 and the estimated detection limits (EDLs) are less than the estimated minimum levels

(EMLs), report the data as is with qualifiers in the report and a discussion in the case narrative. If the S/N is < 10 or the estimated detection limits (EDLs) are greater than the estimated minimum levels (EMLs), re-extract and reanalyze the sample. If the ion chromatogram of the PFK lock mass indicates ion suppression in the region where the internal standard elutes, reanalyzing the extract at up to a 1/10 dilution may improve the internal standard recovery. If the poor internal standard recovery is judged to be a result of sample matrix, a reduced portion of the sample may be re-extracted or additional cleanups may be employed. The decision to reanalyze or flag the data should be made in consultation with the client.

9.2.2 Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.3 Cleanup Standards: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled cleanup standards after extraction but prior to extract cleanup. They are used to assess the efficiency of the cleanup procedures.

9.4 Recovery Standards: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled recovery standards prior to analysis. They are used to measure the recovery of the internal standards and the cleanup standards.

9.5 Method Blanks

9.5.1 A laboratory method blank must be run along with each analytical batch of 20 or fewer samples. The method blank consists of reagent water for aqueous samples, sodium sulfate for solid and tissue samples, processed in the same manner and at the same time as the associated samples. The method blank is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data. Analyze the blank immediately after analysis of the LCS to demonstrate freedom from contamination. The method blank should not contain any of the compounds of interest at a concentration above the estimated minimum level (EML) shown in Table 4.

9.5.2 Corrective action is required when compounds of interest are detected in the method blank above the EML. Corrective action may include reanalysis of the method blank. Contact the Project Manager to determine further corrective action. At a minimum, all associated results are qualified with a B flag. Re-extraction and reanalysis of all samples associated with a contaminated method blank is required if requested by the client or Project Manager. Investigation of the source of the method blank contamination will be initiated before further samples are extracted.

9.5.3 The method blank must have acceptable internal standard recoveries. If internal standard recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If internal standard recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples

will normally be required. Consultation with the client should take place.

9.5.4 Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.6 Instrument Blank

9.6.1 Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed.

9.6.2 An instrument blank consists of solvent with the internal standards and recovery standards added. It is evaluated in the same way as the method blank.

9.7 Laboratory Control Sample – A laboratory control sample (LCS) is prepared and analyzed with every batch of 20 or fewer samples. All analytes must be within established control limits specified in the appropriate Table 10A or 10B. The LCS is spiked with the compounds listed in Table 5a.

9.7.1 If any analyte in the LCS is outside the control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.
- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported and the failure is documented in the project narrative.

9.8 QC Check Sample - Analyze the QC Check Sample (section 7.7) periodically to assure the accuracy of calibration standards and the overall reliability of the analytical process. It is suggested that the QC Check Sample be analyzed at least annually.

10 Calibration and Standardization

10.1 Three types of calibration procedures are required. The first type establishes retention times, relative retention times and relative retention time windows to be used during the subsequent calibrations and analyses. The second type, initial calibration, is required to establish response factors and is required before any samples are analyzed. It may be required intermittently throughout sample analyses as dictated by the results of continuing calibration procedures described below. The third type, continuing calibration, consists of analyzing the continuing calibration verification solution (VER). No samples are to be analyzed until acceptable calibration as described in sections 10.2, 10.3 and 10.4 is demonstrated and documented.

10.2 Retention Time Calibration

Retention time calibration is required if the retention time criteria cannot be met.

10.2.1 The absolute retention time of CB 209 must exceed 55 minutes. Otherwise the GC temperature program must be adjusted and the test repeated until the requirement is met.

10.2.2 **NOTE**: When adjusting chromatographic conditions, the resolution requirements of sections 10.4.5.8 to 10.4.5.9 must be maintained.

10.2.3 Tune the instrument to meet the mass resolution and mass accuracy requirements of section 10.3.2. Document the resolution and accuracy.

10.2.4 Analyze 2µL of each of the five individual PCB mixtures (section 7.6.3). Repeat the series twice more in succession to provide 3 runs of each mix. It is not necessary to interrupt this analytical sequence to perform a 12-hour resolution check. Set the switch-points for the MID descriptors. The switch-points must be set to insure that the first and last eluting isomer of each homolog group and the labeled internal standards are acquired properly. Determine the average retention time of each PCB congener using the elution order information in Table 11.

NOTE 1: PCB Mixture 5 (M-1668A-5) contains the first and last eluting isomer in each homolog group for the SPB-Octyl column (see Table 7).

NOTE 2: Laboratory data has indicated that the SPB-Octyl column can exhibit significant differences in performance from column to column. It has also been indicated that the column's performance can change significantly due to oxidation with subsequent changes in congener retention times and elution order. The individual PCB mixtures should be analyzed whenever the column's performance or specific congeners retention times are in doubt.

10.2.5 Calculate the relative retention times for all native and labeled congeners, using their retention time references from Table 2 (RT Ref). Calculate the relative retention time for each run in which the congener and its retention time reference are present (i.e., three RRTs will be calculated for each native congener. Fifteen RRTs will be calculated for each internal standard.) Use the calculated average retention times for all native and labeled congeners as the RT calibration source in the calculation software.

10.2.6 Calculate the relative retention time window using the absolute retention time windows (RT Window) from Table 2.

$$\text{RRT Limit Low} = \frac{RT_A - (RT_{WIN}/2)}{RT_{IS}}$$

$$\text{RRT Limit High} = \frac{RT_A + (RT_{WIN}/2)}{RT_{IS}}$$

Where:

RT_A = Average retention time of analyte.

RT_{IS} = Average retention time of RT reference.

RT_{WIN} = Absolute RT window in seconds from Table 2.

10.2.7 A single pair of RRT limits is used for all congeners in coeluting set. Use the

RRT Limit Low that was calculated for the first eluting congener, and the RRT Limit High calculated for the last eluting congener (in the coelution set).

10.3 Initial Calibration

Initial calibration is required before any samples are analyzed for PCBs. Initial calibration is also required if any continuing calibration (section 10.4) does not meet the required criteria in section 10.4.5 after routine maintenance.

10.3.1 Prepare multi-level calibration standards containing the compounds and concentrations as specified in Table 6a. Calibration standards should be stored at room temperature and preferably in amber vials. Calibration standard solutions have an expiration date of ten (10) years from date of receipt based on stability of PCBs and history of vendor's recertification of standard lots.

10.3.2 Establish operating parameters for the GC/MS system (suggested operating conditions are displayed in Figure 1 and Figure 2). By using a PFK molecular leak, tune the instrument (see the appropriate instrument manufacturer's operating manual for tuning instructions) to meet the minimum resolving power of 10,000 (10 percent valley) at mass 342.97924 (PFK). For each MID descriptor group, monitor and record the mass resolution and exact masses of three reference peaks covering the mass range of the descriptor (see below). By using peak matching techniques, verify that the deviation between the exact mass and the theoretical mass for each mass monitored is less than 5 ppm. Iteratively adjust operating parameters and tuning values until the mass resolution and mass accuracy criteria are met for each ion. Document the mass resolution and mass accuracy for each of MID group ion sets. Because of the extensive mass range covered in each MID group, it may not be possible to maintain 10,000 resolution throughout the mass range of the MID group. Therefore, resolution must be greater than 8,000 throughout the mass range and must be greater than 10,000 in the center of the mass range for each MID group. The minimum resolution of 10,000 must be met for mass 342.97924.

MID Group 1 PFK ions - 192.9888, 230.98563, 280.98243
MID Group 2 PFK ions - 268.98243, 292.98243, 380.97605
MID Group 3 PFK ions - 342.97924, 380.97605, 430.97285
MID Group 4 PFK ions - 404.97604, 442.97285, 530.96646

10.3.3 Inject a 2 µL aliquot of the CS 0.5 calibration solution.

10.3.3.1 Ion abundance ratios, minimum levels, and signal-to-noise ratios.

10.3.3.1.1 Measure the SICP areas for each congener or congener group, and compute the ion abundance ratios at the exact masses specified in Table 8. Compare the computed ratio to the theoretical ratio given in Table 9.

10.3.3.1.2 All Toxic/LOC and labeled compounds in the CS-0.5 standard must be within the QC limits in Table 9 for their respective

ion abundance ratios; otherwise, the mass spectrometer must be adjusted and this test repeated until the mass ratios fall within the limits specified. If the adjustment alters the resolution of the mass spectrometer, resolution must be verified (Section 10.3.2) prior to repeat of the test.

10.3.3.1.3 The peaks representing the CBs and labeled compounds in the CS-0.5 calibration standard must have signal-to-noise ratios (S/N) ≥ 10; otherwise, the mass spectrometer must be adjusted and this test repeated until the signal to noise criteria are met.

10.3.3.1.4 An exception to the ion abundance ratio and signal to noise ratio requirements is the secondary ion for dichlorinated biphenyls (mass 223.9974). High background from PFK fragments at 223.9974 results in noise levels which exceed 10% of the signal height at levels that are reliably quantifiable.

10.3.4 Analyze 2 µL of each of the other calibration standards.

10.3.4.1 Isomer specificity

10.3.4.1.1 Use the CS-3 calibration standard to evaluate column performance. The toxic isomers must be uniquely resolved from all other congeners. Isomers may be unresolved so long as they have the same toxicity equivalency factor (TEF) and response factor and so long as these unresolved isomers are uniquely resolved from all other congeners. For example, the SPB-Octyl column achieves unique GC resolution of all Toxics except congeners with IUPAC numbers 156 and 157. This isomeric pair is uniquely resolved from all other congeners and these congeners have the same TEF and response factor.

10.3.4.1.2 Evaluate and document the percent valley between PCBs 34 and 23. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.3.4.1.3 Evaluate and document the percent valley between PCBs 187 and 182. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.3.4.1.4 Classify each congener as resolved or as a member of a coelution set. To be documented as resolved, the valleys between any two isomers must be less than 40 percent of the height of the shorter of the two adjacent peaks. Each member of a coelution set is designated with a qualifier in the format of CXXX, where XXX = the lowest numbered congener in the

set. For example, if PCB 156 and PCB 157 coelute, qualify PCB 157 with “C156”.

10.3.4.2 Calculate the RRF of each compound of interest (target analytes, coelution sets, internal standards, cleanup standards, and surrogate standards) vs. the appropriate reference standard (as specified in Table 2) using the following equation:

$$RRF = \frac{As \times Cis}{Ais \times Cs}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest.
Ais = sum of the areas of the quantitation ions of the appropriate reference standard.
Cis = concentration of the appropriate reference standard.
Cs = concentration of the compound of interest.

NOTE: When calculating the RRF for a coelution set, sum the areas of all isomers in the set. Use the resulting RRF for all congeners within the set.

10.3.4.3 Calculate the mean relative response factor (mean RRF) and the percent relative standard deviation (%RSD) of the response factors for each compound of interest in the six calibration standard solutions using the following equations:

$$\overline{RRF}_{n=6} = \frac{1}{n} \times \sum_{i=1}^{n} RF_i$$

$$\%RSD_{n=6} = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RRF}\right)^2}{n-1}} \times \frac{100}{\overline{RRF}}$$

10.3.4.4 Criteria for Acceptable Calibration - The criteria listed below for acceptable calibration must be met before sample analyses are performed. If acceptable initial calibration is not achieved, identify the root cause, perform corrective action, and repeat the initial calibration. If the root cause can be traced to problems with an individual analysis within the calibration series, repeat the individual analysis and recalculate the percent relative standard deviation. If the calibration is acceptable, document the problem and proceed, otherwise repeat the initial calibration.

10.3.4.5 The percent relative standard deviation (%RSD) for the mean relative response factors for the unlabeled native analytes calculated by isotope dilution must not exceed 20 percent. The percent RSD for the mean relative response factors for the unlabeled native analytes calculated by internal standard must not exceed 35 percent. The percent RSD for the mean relative response factors for the labeled standards must not exceed 35

percent.

10.3.5 Analyze 2µL of the Initial Calibration Verification (ICV) Standard in section 7.6.2 after completion of the ICAL prior to sample analysis. Calculate the concentration of the ICV using the RRFs from the CS-3 standard analyzed in section 10.3.4. Calculate the percent difference (%D) between the expected and the calculated ICV concentration using the following formula:

$$\%D = \frac{(C_{Exp} - C_{Calc})}{C_{Exp}} \times 100$$

Where:
C_{Exp} = The expected concentration of the ICV Standard.
C_{Calc} = The calculated concentration of the ICV Standard.

10.3.5.1 The criteria for acceptance of the ICV Standard are as follows:

- The %D may not exceed ±35% for more than 4 of the native and labeled compounds.
- The %D may not exceed ±50% for any native or labeled compound.

10.3.5.2 All data associated with compounds with percent differences exceeding ±35% must be reviewed before acceptance and shall be documented as a NCM. In addition, all data associated with these compounds require corrective action that may include the following:

- Reanalyze the ICV Standard.
- Replace and reanalyze the ICV Standard.
- Evaluate the instrument performance.
- Evaluate the Initial Calibration Standards.

10.4 Continuing Calibration

10.4.1 Continuing calibration is performed at the beginning of a 12-hour period after successful mass resolution check.

10.4.2 Document the mass resolution performance as specified in section 10.3.2 at both the beginning and end of the 12-hour period.

10.4.3 Analyze 2 µL of the Continuing Calibration Verification Standard (VER/CS3). Calculate the concentration (C) of the compounds of interest (target analytes, internal standards, cleanup standards, and surrogate standards) vs. the appropriate quantitation reference (as specified in Table 2) using the following equation:

$$C = \frac{As \times Cis}{Ais \times RRF}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest.
Ais = sum of the areas of the quantitation ions of the appropriate reference standard.
Cis = concentration of the appropriate reference standard.
RRF = mean relative response factor from section 10.3.4.2.

10.4.4 Calculate the concentrations as percentages of the test concentrations and compare them to the limits specified in the appropriate Table 10A or 10B using the following equation:

$$C_{ver}\% = \frac{C_{ver}}{C_{test}} \times 100$$

Where:

C_{ver} = the concentration of the VER standard calculated in section 10.4.3
C_{test} = the test concentration of the VER standard listed in Table 6a.

10.4.5 Criteria for Acceptable Calibration - The criteria listed below for acceptable calibration must be met before sample analyses are performed. If the acceptance criteria are met, the calibration is deemed to be in control and the RRFs generated from the initial calibration are used to quantify samples. If acceptable calibration is not achieved, identify the root cause, perform corrective action, and repeat the continuing calibration. If a second consecutive attempt at a continuing calibration fails, two consecutive calibrations must meet the criteria, or an initial calibration must be run before proceeding with client samples.

10.4.5.1 The ion abundance ratios of the peaks representing the Toxics/LOCs and labeled standards must be within the control limits specified in Table 9.

10.4.5.2 The S/N for the GC signals present in every SICP (including those for labeled standards) must be ≥ 10.

10.4.5.3 For Toxic and LOC congeners, as listed in the first section of Table 10A or 10B, the percent of the calculated concentration relative to the test concentration must be within 70-130% for 1668A and 1668B and 75-125% for 1668C.

10.4.5.4 For non-Toxic congeners the calculated concentrations must be within 70-130% of the test concentrations for 1668A. 1668B and 1668C.

10.4.5.5 The absolute retention times (RT) of the labeled internal standards must be within ±15 seconds of the retention times obtained during initial calibration.

10.4.5.6 The relative retention times (RRT) of the Toxics/LOC congeners must be within their respective RRT limits generated in the retention time calibration in section 10.2.

10.4.5.6.1 If the RRTs or RTs are not within the limits above, the GC may not be performing properly. However, routine column maintenance may include removing short amounts of the

beginning of the column when active sites or non-volatile compounds in sample extracts cause poor chromatography and loss of specificity. Shortening of the column can cause the RRTs or RTs to fall outside the above limits.

10.4.5.6.2 When the RRT of any compound or the RT of any internal standard is not within the above limits, corrective action must be taken. If the GC is not performing properly, correct the problem and repeat the test. If the GC is performing properly but the RRTs or RTs have changed due to routine column maintenance, adjust the GC or replace the GC column, then repeat the test or repeat the retention time calibration.

10.4.5.7 Evaluate and document the percent valley between PCBs 34 and 23. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.4.5.8 Evaluate and document the percent valley between PCBs 187 and 182. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.4.6 Daily calibration must be performed every 12 hours of instrument operation. The 12-hour shift begins with the documentation of the mass resolution followed by the injection of the Continuing Calibration Standard (VER).

11 Procedure

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgement of supervision to accommodate variations in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variations in the procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist, Project Manager, and QA Manager. If contractually required, the client shall be notified.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2 Sample Extraction and Cleanup

The extraction and cleanup procedures are described in SOP KNOX-OP-0021, "Extraction of Polychlorinated Biphenyl (PCB) Isomers for Analysis by Isotope Dilution HRGC/HRMS", current revision.

11.3 Sample Analysis

11.3.1 Analyze the sample extracts under the same instrument operating conditions used to perform the instrument calibrations. Inject 2 µL into the GC/MS and acquire data beginning at 8 minutes and ending after decachlorobiphenyl has eluted from the column.

11.3.2 Record analysis information in the instrument logbook. The following

information is required:

- Date of analysis
- Time of analysis
- Instrument data system filename
- Analyst
- Lab sample identification

Additional information may be recorded in the logbook if necessary.

11.3.3 Generate integrated ion chromatograms for the masses listed in Table 8 that encompass the expected retention windows of the PCB homologous series.

11.3.4 Generate a reduced peak list file from the integrations shown in the ion chromatograms.

11.3.5 Load the reduced peak list file into the calculation software.

11.3.6 The RTs of the unambiguous labeled congeners (RT Markers) are used to calculate a least squares best fit regression for retention times compared to those of the retention time calibration.

11.3.7 The resulting regression is used to calculate predicted retention times for target analytes. These predicted retention times are used by the software to identify candidate peaks for targets.

11.3.8 The analyst reviews the peaks identified as targets and determines whether to accept the identification. This determination is made by evaluating the delta values (RT shift from predicted), knowledge of peak patterns and observations of localized shifting.

11.3.9 A RRT window is calculated by multiplying the RRT Limit High and the RRT Limit Low by the retention time of the designated RT reference. The software applies a qualitative flag to each peak identified as a target that has a RRT outside the RRT window.

11.4 HRGC/HRMS Troubleshooting Guide

11.4.1 Perform the instrument's leak check: Evaluate the air spectrum. Mass 28 should be less than 5 times mass 69. If the air spectrum is not acceptable, replace transfer line ferrule and/or pump out PFK reservoir.

11.4.2 Check the voltage on the 5V power supply: If voltage is not reading 5V, adjust voltage to read 5V.

11.4.3 Check daily calibration standard: Evaluate the signal to noise (S/N), examine peak shape/chromatography, and evaluate response factors. Refer to specific SOP's requirements and acceptance criteria for all natives and internal standards. If daily calibration is not acceptable , perform the following troubleshooting options as needed and reanalyze a daily CS-3 calibration standard:

- Replace the inlet seal and clean the injection port body with methanol to improve response factor.
- Perform column/injector port maintenance and/or retune the instrument to resolve chromatography/resolution, signal to noise issues.
- Perform a gain test on the electron multiplier.
- Adjust the tuning parameters of the instrumentation to achieve optimum sensitivity and peak shape.
- Evaluate carryover from sample analyses. If carryover or contamination is suspected, run solvent rinses (nonane) under MID to evaluate the contamination.
- When troubleshooting cannot resolve an instrument problem, a manufacture's service engineer may be consulted for possible solution, or called onsite for diagnosis/repair.

11.5 Refer to the TestAmerica Knoxville Quality Assurance Manual, KX-QAM, current revision for the HRGC/HRMS instrument equipment maintenance table.

11.6 Refer to TestAmerica Knoxville SOP KNOX-IT-0001, current revision for requirements for computer hardware and software.

12 Data Analysis and Calculations

12.1 Qualitative Identification Criteria for PCBs

For a gas chromatographic peak to be identified as a PCB, it must meet all of the following criteria:

12.1.1 The signals for the two exact masses in Table 8 must be present and must maximize within ±2 seconds.

12.1.2 The signal to noise ratio (S/N) for each GC peak at each exact mass must be greater than or equal to (≥) 2.5. (This requirement does not apply to the secondary ion for dichlorinated biphenyls [mass 223.9974]). High background from PFK fragments at 223.9974 results in noise levels which exceed 10% of the signal height at levels that are reliably quantifiable.

12.1.3 The ratio of the integrated areas of the two exact masses specified in Table 8 must be within the limits in Table 9 . Alternately, the ratios may be within ±15% of the ratio in the midpoint (CS-3) calibration or calibration verification (VER), whichever is most recent.

12.1.4 The relative retention time of the peak for a CB must be within the RRT QC limits calculated in section 10.2.5.

NOTE: For native CBs determined by internal standard quantitation, a given CB congener may fall within more than one RT window and be misidentified unless the

RRT windows are made very narrow, as in Table 2. Therefore, consistency of the RT and RRT with other congeners and the labeled compounds may be required for rigorous congener identification. Retention time regression analysis may be employed for this purpose.

12.1.5 If identification is ambiguous, (i.e., some, but not all of the identification criteria are met for a congener) an experienced analyst must determine the presence or absence of the congener.

12.2 Quantitation for PCBs

12.2.1 Calculate the Internal Standard Recoveries (Ris) relative to the Recovery Standard according to the following equation:

$$Ris = \frac{Ais \times Qrs}{Ars \times RRFis \times Qis} \times 100\%$$

Where:

Ais = sum of the areas of the quantitation ions of the appropriate internal standard
Ars = sum of the areas of the quantitation ions of the recovery standard
Qrs = ng of recovery standard added to extract
Qis = ng of internal standard added to sample
RRFis = mean relative response factor of internal standard obtained during initial calibration

NOTE: In some situations, such as source testing, the extract is split for multiple analyses. In this case, Qrs must be correctly calculated to account for the splitting of extracts before the recovery standard was added.

$$\text{Qrs} = \text{Qrss} \times \text{Split}$$

Where:

Qrs = ng of recovery standard added to extract
Qrss= ng of recovery standard added to the split portion of the extract
Split= split ratio of the extract

12.2.2 Calculate the concentration of individual PCBs according to the following equation:

$$Concentration = \frac{As \times Qis}{Ais \times RRF \times W \times S}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest
Ais = sum of the areas of the quantitation ions of the appropriate internal standard
Qis = ng of internal standard added to sample
RRF = mean relative response factor of compound obtained during initial calibration
W = amount of sample extracted (grams or liters)

S = decimal expression of solids (optional, if results are requested to be reported on dry weight basis)

12.2.3 If reporting results for Total Homolog Groups, calculate the total concentration of all isomers within each homolog group by summing the concentrations of the individual PCB isomers within that homolog group.

12.2.4 If no peaks are present in the region of the ion chromatogram where the compounds of interest are expected to elute, calculate the estimated detection limit (EDL) for that compound according to the following equation:

$$EDL = \frac{N \times 2.5 \times Qis}{His \times RRF \times W \times S}$$

Where:

N = sum of peak to peak noise of quantitation ion signals in the region of the ion chromatogram where the compound of interest is expected to elute
His = sum of peak heights of quantitation ions for appropriate internal standard
Qis = ng of internal standard added to sample
RRF = mean relative response factor of compound obtained during initial calibration
W = amount of sample extracted (grams or liters)
S = decimal expression of solids (optional, if results are requested to be reported on dry weight basis.

Note: do not use **S** if results are to be reported by QuantIMS since it performs all necessary moisture corrections.)

12.2.5 If peaks are present in the region of the ion chromatogram which do not meet the qualitative criteria listed in section 12.1, calculate an Estimated Maximum Possible Concentration (EMPC). Use the equation in section 12.2.2, except that "As" should represent the sum of the area under each mass peak calculated using the theoretical chlorine isotope ratio. The peak selected to calculate the theoretical area should be the one which gives the lower of the two possible results (i.e., the EMPC will always be lower than the result calculated from the uncorrected areas).

12.2.6 If the concentration in the final extract of any PCB isomer exceeds the upper method calibration limits, a dilution of the extract or a re-extraction of a smaller portion of the sample must be performed. Dilutions of up to 1/10 may be performed on the extract. If compound concentrations exceeding the calibration range cannot be brought within the calibration range by a 1/10 dilution, extraction of a smaller aliquot of sample may be performed or the sample may be analyzed by a more appropriate analytical technique such as HRGC/LRMS. Consultation with the client should occur before any re-extraction is performed. The lab may report the measured concentration and indicate that the value exceeds the calibration limit by flagging the results with "E". Consultation with the client should occur before compounds are reported which exceed the calibration range.

12.3 The estimated minimum level (EML) is defined as the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present assuming a sample is extracted at the recommended weight or volume and is carried through all normal extraction and analysis procedures. The EMLs for different matrices and extract volumes are listed in Table 4. Deviations from the extraction amounts or final volumes listed will result in corresponding changes in the actual sample EMLs.

12.4 Flag all compound results in the sample which are below the estimated minimum level with a "J" qualifier.

12.5 Flag all compound results in the sample which were detected in the method blank with a "B" qualifier.

12.6 Flag all compound results in the sample which are above the upper calibration limit with an "E" qualifier.

12.7 Flag all compound results in the sample which are "Estimated Maximum Possible Concentrations" with a "Q" qualifier.

12.8 Flag compound results in the sample that may contain co-eluting compounds with a "C" qualifier.

12.8.1 Flag congeners known to coelute with a higher numbered congener with a "C" qualifier.

12.8.2 Flag congeners that coelute with a lower numbered congener with a "Cx" qualifier where x is the CAS PCB number of the lowest numbered congener in the coeluting group.

12.9 If DOD QSM requirements are used, refer to SOP KNOX-QA-0021.

12.10 Flag compound results in the sample that may be affected by ion suppression with a "S" qualifier. When ion suppression of a PFK trace occurs at greater than or equal to 20% of full scale on both the lock mass and QC mass traces, and when the suppression is sustained for greater than 4 seconds, the suppression must be evaluated to determine which, if any, PCB congeners co-elute with the suppression. Samples may be diluted to decrease the effects of the ion suppression and rerun. Congeners that are determined to co-elute with the suppression are flagged with an "S" qualifier.

12.11 Data Review

12.11.1 Refer to Figure 3 for an example data review checklists used to perform and document the review of the data. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data.

12.11.2 The analyst who performs the initial data calculations must initial and date the front chromatogram of the raw data package to document that they have performed the qualitative and quantitative analysis on the sample data.

12.11.3 A second analyst must verify all qualitative peak identifications. If discrepancies

are found, the data must be returned to the analyst who performed the initial peak identification for resolution.

12.11.4 A second analyst must check all hand calculation and data entry into calculation programs, databases, or spreadsheets at a frequency of 100 percent. If discrepancies are found, the data must be returned to the analyst who performed the initial calculation for resolution.

12.11.5 The reviewing analyst must initial and date the front chromatogram of the raw data package to document that they have performed the second level review on the sample data.

12.11.6 All items listed on the data review checklist must be checked by both the analyst who performed the initial qualitative and quantitative analysis and the analyst who performed the second level review. An example data review checklist is shown in Figure 3.

13 Method Performance

13.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. The procedure for determination of the method detection limit is given in the SOP CA-Q-S006, current revision, based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit (ML).

13.2 Initial Demonstration of Capability – Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in TestAmerica Knoxville SOP KNOX-QA-0009.

13.3 Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009 current revision for further requirements for performing and documenting initial and on-going demonstrations of capability**.**

14 Pollution Prevention

14.1 All attempts will be made to minimize the use of solvents and standard materials.

15 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 Waste Streams Produced by the Procedure: The following waste streams are produced when this method is carried out.

- Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar

materials shall be placed in the incinerable laboratory waste stream, contained in a steel or poly satellite accumulation container.

16 References

16.1 Knoxville Laboratory Quality Assurance Manual (QAM), current revision.

16.2 Method 1668, Revision A, B and C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-00-002, December 1999.

16.3 Method 1613: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS [Revision B], EPA#: 821/B-94-005a YEAR: 1994

16.4 Ballschmiter, K. and M. Zell, "Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography", *Fresenius Z. Anal. Chem*., 302:20-31 (1980).

16.5 Schulte, E. and R. Malisch, "Berechnung der Wahren PCB-Gehalte in Umweltproben I. Ermittlung der Zusammensetzung Zweier Technischer PCB-Gemische," *Fresenius Z. Anal. Chem*., 314:545-551 (1983).

16.6 Guitart, R., P. Puig and J. Gómez-Catalán, "Requirement for a Standardized Nomenclature Criterion for PCBs: Computer-Assisted Assignment of Correct Congener Denomination and Numbering," *Chemosphere*, 27(8):1451-1459 (1993).

16.7 Rigaudy, J. and Klesney, S.P., Nomenclature of Organic Chemistry, Pergamon, 1979.

16.8 Pretsch, Clerc, Seibl, Simon, Tables of Spectral Data for Structure Determination of Organic Compounds, Second Edition, Springer-Verlag, 1989.

16.9 CRC Handbook of Chemistry and Physics, 71st edition, CRC Press, 1990-1991.

16.10 Email dated 03/02/10 from Richard Reding Ph.D., Chief U.S. EPA, Engineering & Analytical Support Branch. Engineering & Analysis Division Office of Science and Technology, Office of Water – Subject: Use of QC Acceptance Limits.”

17 Miscellaneous

17.1 Deviations from EPA Method 1668, Revisions A, B and C.

17.1.1 Additional recovery standards are used in this procedure. The additional standards are listed in Table 1.

17.1.2 Additional labeled standards are used in this procedure as field sampling surrogates. The additional standards are listed in Table 1

17.1.3 This procedure uses internal standards which are within the same MID group as the native congener that is being calculated. This improvement is used to reduce the effects of full verses reduced accelerating voltage tuning differences which can be exaggerated when crossing MID groups.

17.1.4 The calibration procedure in the method 1668A, revision B, and C call for a

single point standard for the non-Toxic/LOC congeners. This procedure uses a multi-point calibration for all 209 congeners.

17.1.5 This procedure uses MID groups that differ from the method. The procedure uses 4 groups, rather than 6, to improve instrument stability, by holding the magnet current steady for longer periods. Therefore alternate PFK lock masses are monitored, to reflect the mass ranges of the procedure's MID groups.

17.1.6 This procedure uses average retention times (and average relative retention times) produced by triplicate analyses of the 5 mixes specified, rather than single analyses of the diluted 209 standard.

17.1.7 The absolute retention times, relative retention times, and relative retention time limits used by the laboratory differ from those listed in all versions of Method 1668. Each SPC-Octyl column lot used by the laboratory has exhibited slightly different retention time characteristics resulting in different absolute retention times than those listed in the method or those observed with another SPB-Octyl column lot. To ensure that the correct peak assignments are made, a retention time study is performed for each new column lot to verify the original study. This study includes the triplicate analysis of the five retention time mixes listed in Method 1668A as described in Section 10.2.5 to 10.2.7. This procedure requires a minimum elution time of 55 minutes for PCB 209.

17.1.8 The calibration verification procedures in the method call for updating the retention times, relative retention times and response factors for non-Toxic compounds during daily calibration and use the retention times, relative retention times and response factors from the initial calibration for Toxic and LOC compounds. This laboratory uses the retention times and relative retention times from triplicate analyses of the 5-mix series, which contains all congeners, and uses response factors from the initial calibration for all 209 compounds. The practice of updating the relative retention times of only a subset of compounds causes significant error in the linear regression prediction formulas used by targeting software to identify the compounds. This procedure has provisions for updating all RTs and RRTs by analyzing a new retention time calibration series.

17.1.9 The EMLs listed in Table 4 differ from those listed in the reference method. The EMLs are set above the mean plus 2 standard deviations for the higher of detections or EDLs for method blanks. In no case is the EML lower than the low calibration limit. The survey period was approximately 14 months.

17.1.10 This procedure uses a 100uL final volume for solid samples, or a variation depending on the protocol assigned to the sample based on screen results prior to extraction.

17.1.11 Method 1668C requires recovery standards be added immediately prior to analysis. In this procedure recovery standards are added to sample extracts at the conclusion of the extraction / clean up process prior to analysis.

17.1.12 Method 1668C implies that all qualitative criteria must be met in order for a

positive hit to be reported. This procedure allows positive hits to be reported with "Q" qualifiers.

17.1.13 This procedure uses calculated concentrations for determining if a dilution is required not area counts as suggested by Method 1668C, section 17.5.

17.1.14 This procedure reports positive hits below the estimated minimum level (EML) which are determined to be greater than 2.5 signal to noise. These hits are qualified with a "J".

17.1.15 This procedure does not report blank corrected values.

17.1.16 When samples are diluted to bring a native analyte within the upper calibration range all 209 congeners are reported from this analysis. This procedure reports all congeners from one analytical analysis.

17.1.17 This procedure uses a daily calibration CCV consisting of all 209 congeners at the beginning of each 12 hour analytical shift.

17.1.18 Extracts are stored in a dark box at room temperature in the same manner as standards.

17.1.19 1668C EML limits are listed in Table 4. However, this laboratory currently uses the 1668A/B limits

17.1.20 The limits established in 1668B, Table 6 for the IPR and OPR performance evaluations have been observed by EPA reviewers to penalize an analyst who achieves better performance than those observed in the inter-laboratory study. Based on EPA's recommendations (section 16.10), TestAmerica Knoxville performs analysis by Method 1668B with the QC acceptance criteria in Table 6 of EPA Method 1668A. Table 10 "Acceptance Criteria for Performance Tests" of this SOP will be used for the IPR and OPR limits.

17.2 List of tables and figures referenced in the body of the SOP.

17.2.1 Table 1 – Polychlorinated Biphenyls Determined by High Resolution Gas Chromatography (HRGC)/High Resolution Mass Spectrometry (HRMS)

17.2.2 Table 2 – RT References, Quantitation References, Retention Times (RT), and Relative Retention Times (RRTs) for the 209 CB congeners on SPB-Octyl

17.2.3 Table 3 – Low Calibration Levels Based on Various Final Extract Volumes

17.2.4 Table 4 – Estimated Minimum Levels EPA 1668A, B & C– Matrix and Concentration

17.2.5 Table 5a – Concentration of Native PCB Congener Stock and Spiking Solutions

17.2.6 Table 5b – Concentration of $^{13}C_{12}$ Labeled PCB Congener Stock and Spiking Solutions

17.2.7 Table 6a – Concentration of PCBs in Calibration Solutions

17.2.8 Table 6b – Preparation of Calibration Solutions

17.2.9 Table 7 – Window Defining Mixture and SPB-Octyl Resolution Test Compounds

17.2.10 Table 8 – Ions Monitored for HRGC/HRMS Analysis of PCBs

17.2.11 Table 9 – Theoretical Ion Abundance Ratios and Control Limits for PCBs

17.2.12 Table 10A – Acceptance Criteria for Performance Tests (1668A & B)

17.2.13 Table 10B – Acceptance Criteria for Performance Tests (1668C)

17.2.14 Table 11 – Retention Times of Isomers on SPB-Octyl Column for PCB Standard Mixes

17.2.15 Table 12 – Assignment of Sample Preparation Protocols

17.2.16 Figure 1 – Recommended GC Operating Conditions

17.2.17 Figure 2 – Recommended MID Descriptors

17.2.18 Figure 3 – Example Data Review Checklist

17.2.19 Table 1 - Polychlorinated Biphenyls Determined by HRGC/HRMS

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	U
1	1	2-monochlorobiphenyl	2051-60-7	$^{13}C_{12}$-2-monochlorobiphenyl	234432-85-0	Inter
2	2	3-monochlorobiphenyl	2051-61-8			
3	3	4-monochlorobiphenyl	2051-62-9	$^{13}C_{12}$-4-monochlorobiphenyl	208263-77-8	Inter
4	4	2,2'-dichlorobiphenyl	13029-08-8	$^{13}C_{12}$-2,2'-dichlorobiphenyl	234432-86-1	Inter
5	5	2,3-dichlorobiphenyl	16605-91-7			
6	6	2,3'-dichlorobiphenyl	25569-80-6			
7	7	2,4-dichlorobiphenyl	33284-50-3			
8	8	2,4'-dichlorobiphenyl	34883-43-7	$^{13}C_{12}$-2,4'-dichlorobiphenyl		Surro
9	9	2,5-dichlorobiphenyl	34883-39-1	$^{13}C_{12}$-2,5-dichlorobiphenyl	250694-89-4	Reco
10	10	2,6-dichlorobiphenyl	33146-45-1			
11	11	3,3'-dichlorobiphenyl	2050-67-1			
12	12	3,4-dichlorobiphenyl	2974-92-7			
13	13	3,4'-dichlorobiphenyl	2974-90-5			
14	14	3,5-dichlorobiphenyl	34883-41-5			
15	15	4,4'-dichlorobiphenyl	2050-68-2	$^{13}C_{12}$-4,4'-dichlorobiphenyl	208263-67-6	Inter
16	16	2,2',3-trichlorobiphenyl	38444-78-9			
17	17	2,2',4-trichlorobiphenyl	37680-66-3			
18	18	2,2',5-trichlorobiphenyl	37680-65-2			
19	19	2,2',6-trichlorobiphenyl	38444-73-4	$^{13}C_{12}$-2,2',6-trichlorobiphenyl	234432-87-2	Inter
20	20	2,3,3'-trichlorobiphenyl	38444-84-7			
21	21	2,3,4-trichlorobiphenyl	55702-46-0			
22	22	2,3,4'-trichlorobiphenyl	38444-85-8			
23	23	2,3,5-trichlorobiphenyl	55720-44-0			
24	24	2,3,6-trichlorobiphenyl	55702-45-9			
25	25	2,3',4-trichlorobiphenyl	55712-37-3			
26	26	2,3',5-trichlorobiphenyl	38444-81-4			
27	27	2,3',6-trichlorobiphenyl	38444-76-7			
28	28	2,4,4'-trichlorobiphenyl	7012-37-5	$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	208263-76-7	Clea
29	29	2,4,5-trichlorobiphenyl	15862-07-4			
30	30	2,4,6-trichlorobiphenyl	35693-92-6			
31	31	2,4',5-trichlorobiphenyl	16606-02-3	$^{13}C_{12}$-2,4',5-trichlorobiphenyl		Reco
32	32	2,4',6-trichlorobiphenyl	38444-77-8	$^{13}C_{12}$-2,4',6-trichlorobiphenyl		Reco
33	33	2',3,4-trichlorobiphenyl (2,3',4'-trichlorobiphenyl)	38444-86-9			
34	34	2',3,5-trichlorobiphenyl (2,3',5'-trichlorobiphenyl)	37680-68-5			
35	35	3,3',4-trichlorobiphenyl	37680-69-6			
36	36	3,3',5-trichlorobiphenyl	38444-87-0			
37	37	3,4,4'-trichlorobiphenyl	38444-90-5	$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	208263-79-0	Inter
38	38	3,4,5-trichlorobiphenyl	53555-66-1			
39	39	3,4',5-trichlorobiphenyl	38444-88-1			
40	40	2,2',3,3'-tetrachlorobiphenyl	38444-93-8			
41	41	2,2',3,4-tetrachlorobiphenyl	52663-59-9			
42	42	2,2',3,4'-tetrachlorobiphenyl	36559-22-5			
43	43	2,2',3,5-tetrachlorobiphenyl	70362-46-8			
44	44	2,2',3,5'-tetrachlorobiphenyl	41464-39-5			
45	45	2,2',3,6-tetrachlorobiphenyl	70362-45-7			
46	46	2,2',3,6'-tetrachlorobiphenyl	41464-47-5			
47	47	2,2',4,4'-tetrachlorobiphenyl	2437-79-8			
48	48	2,2',4,5-tetrachlorobiphenyl	70362-47-9			
49	49	2,2',4,5'-tetrachlorobiphenyl	41464-40-8			
50	50	2,2',4,6-tetrachlorobiphenyl	62796-65-0			
51	51	2,2',4,6'-tetrachlorobiphenyl	68194-04-7			
52	52	2,2',5,5'-tetrachlorobiphenyl	35693-99-3	$^{13}C_{12}$-2,2',5,5'-tetrachlorobiphenyl	160901-66-6	Reco
53	53	2,2',5,6'-tetrachlorobiphenyl	41464-41-9			
54	54	2,2',6,6'-tetrachlorobiphenyl	15968-05-5	$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	234432-88-3	Inter
55	55	2,3,3',4-tetrachlorobiphenyl	74338-24-2			
56	56	2,3,3',4'-tetrachlorobiphenyl	41464-43-1			
57	57	2,3,3',5-tetrachlorobiphenyl	70424-67-8			
58	58	2,3,3',5'-tetrachlorobiphenyl	41464-49-7			
59	59	2,3,3',6-tetrachlorobiphenyl	74472-33-6			
60	60	2,3,4,4'-tetrachlorobiphenyl	33025-41-1			

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	U
61	61	2,3,4,5-tetrachlorobiphenyl	33284-53-6			
62	62	2,3,4,6-tetrachlorobiphenyl	54230-22-7			
63	63	2,3,4’,5-tetrachlorobiphenyl	74472-34-7			
64	64	2,3,4’,6-tetrachlorobiphenyl	52663-58-8			
65	65	2,3,5,6-tetrachlorobiphenyl	33284-54-7			
66	66	2,3’,4,4’-tetrachlorobiphenyl	32598-10-0			
67	67	2,3’,4,5-tetrachlorobiphenyl	73575-53-8			
68	68	2,3’,4,5’-tetrachlorobiphenyl	73575-52-7			
69	69	2,3’,4,6-tetrachlorobiphenyl	60233-24-1			
70	70	2,3’,4’,5-tetrachlorobiphenyl	32598-11-1			
71	71	2,3’,4’,6-tetrachlorobiphenyl	41464-46-4			
72	72	2,3’,5,5’-tetrachlorobiphenyl	41464-42-0			
73	73	2,3’,5’,6-tetrachlorobiphenyl	74338-23-1			
74	74	2,4,4’,5-tetrachlorobiphenyl	32690-93-0			
75	75	2,4,4’,6-tetrachlorobiphenyl	32598-12-2			
76	76	2’,3,4,5-tetrachlorobiphenyl (2,3’,4’,5’-tetrachlorobiphenyl)	70362-48-0			
77	77	3,3’,4,4’-tetrachlorobiphenyl	32598-13-3	$^{13}C_{12}$-3,3’,4,4’-tetrachlorobiphenyl	160901-67-7	Inter
78	78	3,3’,4,5-tetrachlorobiphenyl	70362-49-1			
79	79	3,3’,4,5’-tetrachlorobiphenyl	41464-48-6	$^{13}C_{12}$-3,3’,4,5’-tetrachlorobiphenyl		Surro
80	80	3,3’,5,5’-tetrachlorobiphenyl	33284-52-5			
81	81	3,4,4’,5-tetrachlorobiphenyl	70362-50-4	$^{13}C_{12}$-3,4,4’,5-tetrachlorobiphenyl	160901-68-8	Inter
82	82	2,2’,3,3’,4-pentachlorobiphenyl	52663-62-4			
83	83	2,2’,3,3’,5-pentachlorobiphenyl	60145-20-2			
84	84	2,2’,3,3’,6-pentachlorobiphenyl	52663-60-2			
85	85	2,2’,3,4,4’-pentachlorobiphenyl	65510-45-4			
86	86	2,2’,3,4,5-pentachlorobiphenyl	55312-69-1			
87	87	2,2’,3,4,5’-pentachlorobiphenyl	38380-02-8			
88	88	2,2’,3,4,6-pentachlorobiphenyl	55215-17-3			
89	89	2,2’,3,4,6’-pentachlorobiphenyl	73575-57-2			
90	90	2,2’,3,4’,5-pentachlorobiphenyl	68194-07-0			
91	91	2,2’,3,4’,6-pentachlorobiphenyl	68194-05-8			
92	92	2,2’,3,5,5’-pentachlorobiphenyl	52663-61-3			
93	93	2,2’,3,5,6-pentachlorobiphenyl	73575-56-1			
94	94	2,2’,3,5,6’-pentachlorobiphenyl	73575-55-0			
95	95	2,2’,3,5’,6-pentachlorobiphenyl	38379-99-6	$^{13}C_{12}$-2,2’,3,5’,6-pentachlorobiphenyl		Surro
96	96	2,2’,3,6,6’-pentachlorobiphenyl	73575-54-9			
97	97	2,2’,3’,4,5-pentachlorobiphenyl (2,2’,3,4’,5’-pentachlorobiphenyl)	41464-51-1			
98	98	2,2’,3’,4,6-pentachlorobiphenyl (2,2’,3,4’,6’-pentachlorobiphenyl)	60233-25-2			
99	99	2,2’,4,4’,5-pentachlorobiphenyl	38380-01-7			
100	100	2,2’,4,4’,6-pentachlorobiphenyl	39485-83-1			
101	101	2,2’,4,5,5’-pentachlorobiphenyl	37680-73-2	$^{13}C_{12}$-2,2’,4,5,5’-pentachlorobiphenyl	160901-69-9	Reco
102	102	2,2’,4,5,6’-pentachlorobiphenyl	68194-06-9			
103	103	2,2’,4,5’,6-pentachlorobiphenyl	60145-21-3			
104	104	2,2’,4,6,6’-pentachlorobiphenyl	56558-16-8	$^{13}C_{12}$-2,2’,4,6,6’-pentachlorobiphenyl	234432-89-4	Inter
105	105	2,3,3’,4,4’-pentachlorobiphenyl	32598-14-4	$^{13}C_{12}$-2,3,3’,4,4’-pentachlorobiphenyl	160901-70-2	Inter
106	106	2,3,3’,4,5-pentachlorobiphenyl	70424-69-0			
107	107/109	2,3,3',4',5-pentachlorobiphenyl	70424-68-9			
108	108/107	2,3,3',4,5'-pentachlorobiphenyl	70362-41-3			
109	109/108	2,3,3',4,6-pentachlorobiphenyl	74472-35-8			
110	110	2,3,3’,4’,6-pentachlorobiphenyl	38380-03-9			
111	111	2,3,3’,5,5’-pentachlorobiphenyl	39635-32-0	$^{13}C_{12}$-2,3,3’,5,5’-pentachlorobiphenyl	160901-71-3	Clea
112	112	2,3,3’,5,6-pentachlorobiphenyl	74472-36-9			
113	113	2,3,3’,5’,6-pentachlorobiphenyl	68194-10-5			
114	114	2,3,4,4’,5-pentachlorobiphenyl	74472-37-0	$^{13}C_{12}$-2,3,4,4’,5-pentachlorobiphenyl	160901-72-4	Inter
115	115	2,3,4,4’,6-pentachlorobiphenyl	74472-38-1			
116	116	2,3,4,5,6-pentachlorobiphenyl	18259-05-7			
117	117	2,3,4’,5,6-pentachlorobiphenyl	68194-11-6			
118	118	2,3’,4,4’,5-pentachlorobiphenyl	31508-00-6	$^{13}C_{12}$-2,3’,4,4’,5-pentachlorobiphenyl	160901-73-5	Inter
119	119	2,3’,4,4’,6-pentachlorobiphenyl	56558-17-9			
120	120	2,3’,4,5,5’-pentachlorobiphenyl	68194-12-7			
121	121	2,3’,4,5’,6-pentachlorobiphenyl	56558-18-0			

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	U
122	122	2',3,3',4,5-pentachlorobiphenyl (2,3,3',4',5'-pentachlorobiphenyl)	76842-07-4			
123	123	2',3,4,4',5-pentachlorobiphenyl (2,3',4,4',5'-pentachlorobiphenyl)	65510-44-3	$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	160901-74-6	Inter
124	124	2',3,4,5,5'-pentachlorobiphenyl (2,3',4',5',5-pentachlorobiphenyl)	70424-70-3			
125	125	2',3,4,5,6'-pentachlorobiphenyl (2,3',4',5',6-pentachlorobiphenyl)	74472-39-2			
126	126	3,3',4,4',5-pentachlorobiphenyl	57465-28-8	$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	160901-75-7	Inter
127	127	3,3',4,5,5'-pentachlorobiphenyl	39635-33-1	$^{13}C_{12}$-3,3',4,5,5'-pentachlorobiphenyl		Reco
128	128	2,2',3,3',4,4'-hexachlorobiphenyl	38380-07-3			
129	129	2,2',3,3',4,5-hexachlorobiphenyl	55215-18-4			
130	130	2,2',3,3',4,5'-hexachlorobiphenyl	52663-66-8			
131	131	2,2',3,3',4,6-hexachlorobiphenyl	61798-70-7			
132	132	2,2',3,3',4,6'-hexachlorobiphenyl	38380-05-1			
133	133	2,2',3,3',5,5'-hexachlorobiphenyl	35694-04-3			
134	134	2,2',3,3',5,6-hexachlorobiphenyl	52704-70-8			
135	135	2,2',3,3',5,6'-hexachlorobiphenyl	52744-13-5			
136	136	2,2',3,3',6,6'-hexachlorobiphenyl	38411-22-2			
137	137	2,2',3,4,4',5-hexachlorobiphenyl	35694-06-5			
138	138	2,2',3,4,4',5'-hexachlorobiphenyl	35065-28-2	$^{13}C_{12}$-2,2',3,4,4',5'-hexachlorobiphenyl	160901-76-8	Reco
139	139	2,2',3,4,4',6-hexachlorobiphenyl	56030-56-9			
140	140	2,2',3,4,4',6'-hexachlorobiphenyl	59291-64-4			
141	141	2,2',3,4,5,5'-hexachlorobiphenyl	52712-04-6			
142	142	2,2',3,4,5,6-hexachlorobiphenyl	41411-61-4			
143	143	2,2',3,4,5,6'-hexachlorobiphenyl	68194-15-0			
144	144	2,2',3,4,5',6-hexachlorobiphenyl	68194-14-9			
145	145	2,2',3,4,6,6'-hexachlorobiphenyl	74472-40-5			
146	146	2,2',3,4',5,5'-hexachlorobiphenyl	51908-16-8			
147	147	2,2',3,4',5,6-hexachlorobiphenyl	68194-13-8			
148	148	2,2',3,4',5,6'-hexachlorobiphenyl	74472-41-6			
149	149	2,2',3,4',5',6-hexachlorobiphenyl	38380-04-0			
150	150	2,2',3,4',6,6'-hexachlorobiphenyl	68194-08-1			
151	151	2,2',3,5,5',6-hexachlorobiphenyl	52663-63-5			
152	152	2,2',3,5,6,6'-hexachlorobiphenyl	68194-09-2			
153	153	2,2',4,4',5,5'-hexachlorobiphenyl	35065-27-1	$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl		Surro
154	154	2,2',4,4',5,6'-hexachlorobiphenyl	60145-22-4			
155	155	2,2',4,4',6,6'-hexachlorobiphenyl	33979-03-2	$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	234432-90-7	Inter
156	156	2,3,3',4,4',5-hexachlorobiphenyl	38380-08-4	$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	160901-77-9	Inter
157	157	2,3,3',4,4',5'-hexachlorobiphenyl	69782-90-7	$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	160901-78-0	Inter
158	158	2,3,3',4,4',6-hexachlorobiphenyl	74472-42-7			
159	159	2,3,3',4,5,5'-hexachlorobiphenyl	39635-35-3			
160	160	2,3,3',4,5,6-hexachlorobiphenyl	41411-62-5			
161	161	2,3,3',4,5',6-hexachlorobiphenyl	74472-43-8			
162	162	2,3,3',4',5,5'-hexachlorobiphenyl	39635-34-2			
163	163	2,3,3',4',5,6-hexachlorobiphenyl	74472-44-9			
164	164	2,3,3',4',5',6-hexachlorobiphenyl	74472-45-0			
165	165	2,3,3',5,5',6-hexachlorobiphenyl	74472-46-1			
166	166	2,3,4,4',5,6-hexachlorobiphenyl	41411-63-6			
167	167	2,3',4,4',5,5'-hexachlorobiphenyl	52663-72-6	$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	161627-18-5	Inter
168	168	2,3',4,4',5',6-hexachlorobiphenyl	59291-65-5			
169	169	3,3',4,4',5,5'-hexachlorobiphenyl	32774-16-6	$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	160901-79-1	Inter
170	170	2,2',3,3',4,4',5-heptachlorobiphenyl	35065-30-6	$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	160901-80-4	Inter
171	171	2,2',3,3',4,4',6-heptachlorobiphenyl	52663-71-5			
172	172	2,2',3,3',4,5,5'-heptachlorobiphenyl	52663-74-8			
173	173	2,2',3,3',4,5,6-heptachlorobiphenyl	68194-16-1			
174	174	2,2',3,3',4,5,6'-heptachlorobiphenyl	38411-25-5			
175	175	2,2',3,3',4,5',6-heptachlorobiphenyl	40186-70-7			
176	176	2,2',3,3',4,6,6'-heptachlorobiphenyl	52663-65-7			
177	177	2,2',3,3',4',5,6-heptachlorobiphenyl (2,2',3,3',4,5',6'-heptachlorobiphenyl)	52663-70-4			
178	178	2,2',3,3',5,5',6-heptachlorobiphenyl	52663-67-9	$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	160901-81-5	Clea
179	179	2,2',3,3',5,6,6'-heptachlorobiphenyl	52663-64-6			
180	180	2,2',3,4,4',5,5'-heptachlorobiphenyl	35065-29-3	$^{13}C_{12}$-2,2',3,4,4',5,5'-heptachlorobiphenyl	160901-82-6	Reco

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	U
181	181	2,2',3,4,4',5,6-heptachlorobiphenyl	74472-47-2			
182	182	2,2',3,4,4',5,6'-heptachlorobiphenyl	60145-23-5			
183	183	2,2',3,4,4',5',6-heptachlorobiphenyl	52663-69-1			
184	184	2,2',3,4,4',6,6'-heptachlorobiphenyl	74472-48-3			
185	185	2,2',3,4,5,5',6-heptachlorobiphenyl	52712-05-7			
186	186	2,2',3,4,5,6,6'-heptachlorobiphenyl	74472-49-4			
187	187	2,2',3,4',5,5',6-heptachlorobiphenyl	52663-68-0			
188	188	2,2',3,4',5,6,6'-heptachlorobiphenyl	74487-85-7	$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	234432-91-8	Inter
189	189	2,3,3',4,4',5,5'-heptachlorobiphenyl	39635-31-9	$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	160901-83-7	Inter
190	190	2,3,3',4,4',5,6-heptachlorobiphenyl	41411-64-7			
191	191	2,3,3',4,4',5',6-heptachlorobiphenyl	74472-50-7			
192	192	2,3,3',4,5,5',6-heptachlorobiphenyl	74472-51-8			
193	193	2,3,3',4',5,5',6-heptachlorobiphenyl	69782-91-8			
194	194	2,2',3,3',4,4',5,5'-octachlorobiphenyl	35694-08-7	$^{13}C_{12}$-2,2',3,3',4,4',5,5'-octachlorobiphenyl	208263-74-5	Reco
195	195	2,2',3,3',4,4',5,6-octachlorobiphenyl	52663-78-2			
196	196	2,2',3,3',4,4',5,6'-octachlorobiphenyl	42740-50-1			
197	197	2,2',3,3',4,4',6,6'-octachlorobiphenyl	33091-17-7			
198	198	2,2',3,3',4,5,5',6-octachlorobiphenyl	68194-17-2			
199	201/199	2,2',3,3',4,5,5',6'-octachlorobiphenyl	52663-75-9			
200	199/200	2,2',3,3',4,5,6,6'-octachlorobiphenyl	52663-73-7			
201	200/201	2,2',3,3',4,5',6,6'-octachlorobiphenyl	40186-71-8			
202	202	2,2',3,3',5,5',6,6'-octachlorobiphenyl	2136-99-4	$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	105600-26-8	Inter
203	203	2,2',3,4,4',5,5',6-octachlorobiphenyl	52663-76-0			
204	204	2,2',3,4,4',5,6,6'-octachlorobiphenyl	74472-52-9			
205	205	2,3,3',4,4',5,5',6-octachlorobiphenyl	74472-53-0	$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	234446-64-1	Inter
206	206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	40186-72-9	$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	208263-75-6	Inter
207	207	2,2',3,3',4,4',5,6,6'-nonachlorobiphenyl	52663-79-3			
208	208	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	52663-77-1	$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	234432-92-9	Inter
209	209	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	2051-24-3	$^{13}C_{12}$-decachlorobiphenyl	160901-84-8	Inter

1. The PCB congener number is from Method 1668C and Chemical Abstract Services.
2. The BZ number is from Ballschmiter and Zell (1980). The IUPAC number, when different from the BZ, follows the recommended changes to the BZ number per Schulte and Malisch (1983) and Guitart et al. (1993).
3. The chemical structure names are from Ballschmiter and Zell (1980). IUPAC nomenclature structure names are listed in parenthesis when different from the BZ name (source CAS Registry).
4. Chemical Abstract Service Registry number (source CAS Registry and 1668A Table 1).

Table 2 - RT References, Quantitation References, Retention Times (RTs), and Relative Retention Times (RRTs) for the 209 CB congeners on SPB-Octyl

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
1	1L	9L	9L	30
1	3L	9L	9L	30
1	1	1L	1L	10
1	2	3L	1L/3L	6
1	3	3L	3L	6
2	4L	9L	9L	30
2	9L	9L	9L	25
2	8L	4L	4L/15L	6
2	15L	9L	9L	20
2	4	4L	4L	10
2	10	4L	4L/15L	6
2	9	4L	4L/15L	6
2	7	4L	4L/15L	6
2	6	4L	4L/15L	6
2	5	4L	4L/15L	6
2	8	4L	4L/15L	6
2	14	15L	4L/15L	6
2	11	15L	4L/15L	6
2	13	15L	4L/15L	6
2	12	15L	4L/15L	6
2	15	15L	15L	10
3	19L	32L	32L	30
3	32L	32L	32L	6
3	31L	31L	31L	6
3	28L	31L	31L	20
3	37L	31L	31L	30
3	19	19L	19L	10
3	30	19L	19L	6
3	18	19L	19L	6
3	17	19L	19L	6
3	27	19L	19L	6
3	24	19L	19L	6
3	16	19L	19L	6
3	32	19L	19L	6
3	34	19L	37L	6
3	23	19L	37L	6
3	29	19L	37L	10
3	26	19L	37L	10
3	25	37L	37L	6
3	31	37L	37L	6
3	28	37L	37L	10
3	20	37L	37L	10
3	21	37L	37L	10
3	33	37L	37L	10
3	22	37L	37L	6
3	36	37L	37L	6
3	39	37L	37L	6
3	38	37L	37L	6
3	35	37L	37L	6
3	37	37L	37L	6
4	54L	52L	32L	20
4	52L	52L	52L	25
4	79L	81L	81L/77L	6
4	81L	52L	52L	20
4	77L	52L	52L	20
4	54	54L	54L	10
4	50	54L	81L/77L	10
4	53	54L	81L/77L	10
4	45	54L	81L/77L	10
4	51	54L	81L/77L	10
4	46	54L	81L/77L	6
4	52	54L	81L/77L	6
4	73	54L	81L/77L	6
4	43	54L	81L/77L	6
4	69	54L	81L/77L	10
4	49	54L	81L/77L	10
4	48	54L	81L/77L	6
4	65	54L	81L/77L	10
4	47	54L	81L/77L	10
4	44	54L	81L/77L	10
4	62	54L	81L/77L	10
4	75	54L	81L/77L	10

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
4	59	54L	81L/77L	10
4	42	54L	81L/77L	6
4	41	54L	81L/77L	10
4	71	54L	81L/77L	10
4	40	54L	81L/77L	10
4	64	54L	81L/77L	6
4	72	81L	81L/77L	6
4	68	81L	81L/77L	6
4	57	81L	81L/77L	6
4	58	81L	81L/77L	6
4	67	81L	81L/77L	6
4	63	81L	81L/77L	6
4	61	81L	81L/77L	12
4	70	81L	81L/77L	12
4	76	81L	81L/77L	12
4	74	81L	81L/77L	10
4	66	81L	81L/77L	6
4	55	81L	81L/77L	6
4	56	81L	81L/77L	6
4	60	81L	81L/77L	6
4	80	81L	81L/77L	6
4	79	81L	81L/77L	6
4	78	81L	81L/77L	6
4	81	81L	81L	6
4	77	77L	77L	6
5	104L	101L	101L	20
5	95L	104L	104L	10
5	101L	101L	101L	25
5	111L	101L	101L	20
5	123L	101L	127L	20
5	118L	101L	127L	20
5	114L	101L	127L	20
5	105L	101L	127L	20
5	127L	127L	127L	25
5	126L	101L	127L	20
5	104	104L	104L	10
5	96	104L	104L	10
5	103	104L	104L	6
5	94	104L	104L	6
5	95	104L	104L	10
5	100	104L	104L	10
5	93	104L	104L	10
5	102	104L	104L	10
5	98	104L	104L	10
5	88	104L	104L	12
5	91	104L	104L	10
5	84	104L	104L	6
5	89	104L	104L	6
5	121	104L	104L	6
5	92	123L	104L	6
5	113	104L	104L	10
5	90	104L	104L	10
5	101	104L	104L	10
5	83	104L	104L	12
5	99	104L	104L	10
5	112	104L	104L	6
5	119	104L	104L	16
5	108 (109)	104L	104L	16
5	86	104L	104L	16
5	97	104L	104L	16
5	125	104L	104L	16
5	87	104L	104L	10
5	117	104L	104L	12
5	116	104L	104L	12
5	85	104L	104L	10
5	110	104L	104L	10
5	115	104L	104L	10
5	82	104L	104L	6
5	111	104L	104L	6
5	120	104L	104L	6
5	107 (108)	104L	123L/114L/118L/105L/126L	10
5	124	104L	123L/114L/118L/105L/126L	10
5	109 (107)	104L	123L/114L/118L/105L/126L	6
5	123	123L	123L	6
5	106	123L	123L/114L/118L/105L/126L	6
5	118	118L	118L	6
5	122	118L	123L/114L/118L/105L/126L	6

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
5	114	114L	114L	6
5	105	105L	105L	6
5	127	105L	123L/114L/118L/105L/126L	6
5	126	126L	126L	6
6	155L	138L	101L	20
6	153L	167L	156L/157L/167L/169L	10
6	138L	138L	138L	100
6	167L	138L	138L	20
6	156L	138L	138L	6
6	157L	138L	138L	20
6	169L	138L	138L	6
6	155	155L	155L	10
6	152	155L	155L	6
6	150	155L	155L	6
6	136	155L	155L	6
6	145	155L	155L	6
6	148	155L	155L	6
6	151	155L	155L	10
6	135	155L	155L	10
6	154	155L	155L	10
6	144	155L	155L	6
6	147	155L	156L/157L/167L/169L	10
6	149	155L	156L/157L/167L/169L	10
6	134	155L	156L/157L/167L/169L	10
6	143	155L	156L/157L/167L/169L	10
6	139	155L	156L/157L/167L/169L	10
6	140	155L	156L/157L/167L/169L	10
6	131	155L	156L/157L/167L/169L	6
6	142	155L	156L/157L/167L/169L	6
6	132	155L	156L/157L/167L/169L	10
6	133	155L	156L/157L/167L/169L	6
6	165	167L	156L/157L/167L/169L	6
6	146	167L	156L/157L/167L/169L	6
6	161	167L	156L/157L/167L/169L	6
6	153	167L	156L/157L/167L/169L	10
6	168	167L	156L/157L/167L/169L	10
6	141	167L	156L/157L/167L/169L	6
6	130	167L	156L/157L/167L/169L	6
6	137	167L	156L/157L/167L/169L	6
6	164	167L	156L/157L/167L/169L	6
6	138	167L	156L/157L/167L/169L	14
6	163	167L	156L/157L/167L/169L	14
6	129	167L	156L/157L/167L/169L	14
6	160	167L	156L/157L/167L/169L	10
6	158	167L	156L/157L/167L/169L	6
6	166	167L	156L/157L/167L/169L	10
6	128	167L	156L/157L/167L/169L	10
6	159	167L	156L/157L/167L/169L	6
6	162	167L	156L/157L/167L/169L	6
6	167	167L	167L	6
6	156	156L	156L/157L	6
6	157	157L	156L/157L	10
6	169	169L	169L	6
7	188L	180L	180L	20
7	178L	180L	180L	20
7	180L	180L	180L	100
7	170L	180L	180L	20
7	189L	180L	194L	20
7	188	188L	188L	6
7	179	188L	188L/170L	6
7	184	188L	188L/170L	6
7	176	188L	188L/170L	6
7	186	188L	188L/170L	6
7	178	188L	188L/170L	6
7	175	188L	188L/170L	6
7	187	188L	188L/170L	6
7	182	188L	188L/170L	6
7	183	188L	188L/170L	6
7	185	188L	188L/170L	6
7	174	188L	188L/170L	6
7	177	188L	188L/170L	6
7	181	188L	188L/170L	6
7	171	188L	188L/170L	10
7	173	188L	188L/170L	6
7	172	189L	188L/170L	6
7	192	189L	188L/170L	6
7	193	189L	188L/170L	6

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
7	180	189L	188L/170L	6
7	191	189L	188L/170L	6
7	170	189L	170L	6
7	190	189L	188L/170L	6
7	189	189L	189L	6
8	202L	194L	180L	20
8	194L	194L	194L	25
8	205L	194L	194L	30
8	202	202L	202L	10
8	201	202L	202L	6
8	204	202L	202L	6
8	197	202L	202L	6
8	200	202L	202L	6
8	198	202L	202L	10
8	199	202L	202L	6
8	196	205L	202L	6
8	203	205L	202L	6
8	195	205L	205L	6
8	194	205L	205L	6
8	205	205L	205L	6
9	208L	194L	194L	20
9	206L	194L	194L	30
9	208	208L	208L	6
9	207	208L	208L/206L	6
9	206	206L	206L	6
10	209L	194L	194L	30
10	209	209L	209L	6

1. Number of chlorines on congener.
2. Suffix "L" indicates labeled compound.
3. IUPAC Number per Table 2 of Method 1668A. (Numbers in parentheses are PCB Numbers from 1668C)
4. Retention time reference that is used to locate target congener.
5. Quantitation reference that is used to calculate the concentration of the target congener or labeled standard.
6. RT window width for congener or group of two or more congeners.

Table 3 - Low Calibration Levels (LCLs) Based on Final Extract Volumes

		20 µL Extract Volume	100µL Extract Volume
Analyte	Minimum Cal. Level CS 0.5 (ng/mL)	Water 1L (ng/L)	Solids and Tissues 10g[2] (ng/g)
Monochlorobiphenyls	0.5	0.01[1]	0.005
Dichlorobiphenyls	0.5	0.01[1]	0.005
Trichlorobiphenyls	0.5	0.01	0.005
Tetrachlorobiphenyls	0.5	0.01	0.005
Pentachlorobiphenyls	0.5	0.01	0.005
Hexachlorobiphenyls	0.5	0.01	0.005
Heptachlorobiphenyls	0.5	0.01	0.005
Octachlorobiphenyls	0.5	0.01	0.005
Nonachlorobiphenyls	0.5	0.01	0.005
Decachlorobiphenyl	0.5	0.01	0.005

1. This value reflects the LCL. Reliable detection at this level may not be attained due to evaporative loss in adjusting the extract volume to 20 µL for these homolog groups.

2. The values for solids and tissues reflect the LCLs for Protocol 1 as described in Table 12. If the sample is prepared by another protocol described in that table, the LCLs shown in this table must be adjusted appropriately.

Table 4 - Estimated Minimum Levels EPA 1668A, B & C- Matrix and Concentration

	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
Parameter	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**
PCB 1	0.040	0.01	1.0	0.02	0.002	1
PCB 2	0.040	0.01	1.0	0.02	0.002	1
PCB 3	0.040	0.01	1.0	0.05	0.005	2.5
PCB 4	0.060	0.02	2.0	0.05	0.005	2.5
PCB 5	0.040	0.01	1.0	0.02	0.002	1
PCB 6	0.040	0.01	1.0	0.02	0.002	1
PCB 7	0.040	0.01	1.0	0.02	0.002	1
PCB 8	0.060	0.02	2.0	0.05	0.005	2.5
PCB 9	0.040	0.01	1.0	0.02	0.002	1
PCB 10	0.040	0.01	1.0	0.05	0.005	2.5
PCB 11	0.060	0.02	2.0	0.10	0.01	5
PCB 12	0.060	0.01	1.0	0.05	0.005	2.5
PCB 14	0.040	0.01	1.0	0.02	0.002	1
PCB 13	0.060	0.01	1.0	0.05	0.005	2.5
PCB 15	0.040	0.01	1.0	0.05	0.005	2.5
PCB 16	0.040	0.01	1.0	0.02	0.002	1
PCB 17	0.040	0.01	1.0	0.02	0.002	1
PCB 18	0.060	0.02	2.0	0.05	0.005	2.5
PCB 30	0.060	0.02	2.0	0.05	0.005	2.5
PCB 19	0.040	0.01	1.0	0.02	0.002	1
PCB 20	0.040	0.02	2.0	0.05	0.005	2.5
PCB 28	0.040	0.02	2.0	0.05	0.005	2.5
PCB 21	0.040	0.01	1.0	0.05	0.005	2.5
PCB33	0.040	0.01	1.0	0.05	0.005	2.5
PCB 22	0.040	0.01	1.0	0.02	0.002	1
PCB 23	0.040	0.01	1.0	0.02	0.002	1
PCB 24	0.040	0.01	1.0	0.02	0.002	1
PCB 25	0.040	0.01	1.0	0.02	0.002	1
PCB 26	0.040	0.01	1.0	0.05	0.005	2.5
PCB 29	0.040	0.01	1.0	0.05	0.005	2.5
PCB 27	0.040	0.01	1.0	0.02	0.002	1
PCB 31	0.040	0.02	2.0	0.05	0.005	2.5
PCB 32	0.040	0.01	1.0	0.02	0.002	1
PCB 34	0.040	0.01	1.0	0.02	0.002	1
PCB 35	0.040	0.01	1.0	0.02	0.002	1
PCB 36	0.040	0.01	1.0	0.02	0.002	1
PCB 37	0.040	0.01	1.0	0.02	0.002	1
PCB 38	0.040	0.01	1.0	0.02	0.002	1
PCB 39	0.040	0.01	1.0	0.02	0.002	1
PCB 40	0.040	0.01	1.0	0.1	0.01	5
PCB 41	0.040	0.01	1.0	0.1	0.01	5
PCB 71	0.040	0.01	1.0	0.1	0.01	5
PCB 42	0.040	0.01	1.0	0.05	0.005	2.5
PCB 43	0.040	0.01	1.0	0.05	0.005	2.5
PCB 73	0.040	0.01	1.0	0.05	0.005	2.5
PCB 44	0.040	0.01	1.0	0.1	0.01	5
PCB 47	0.040	0.01	1.0	0.1	0.01	5
PCB 65	0.040	0.01	1.0	0.1	0.01	5
PCB 45	0.040	0.01	1.0	0.05	0.005	2.5
PCB 51	0.040	0.01	1.0	0.05	0.005	2.5
PCB 46	0.040	0.01	1.0	0.02	0.002	1
PCB 48	0.040	0.01	1.0	0.05	0.005	2.5
PCB 49	0.040	0.01	1.0	0.1	0.01	5
PCB 69	0.040	0.01	1.0	0.1	0.01	5
PCB 50	0.040	0.01	1.0	0.1	0.01	5
PCB 53	0.040	0.01	1.0	0.1	0.01	5
PCB 52	0.040	0.01	1.0	0.05	0.005	2.5

	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
Parameter	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**
PCB 54	0.040	0.01	1.0	0.05	0.005	2.5
PCB 55	0.040	0.01	1.0	0.05	0.005	2.5
PCB 56	0.040	0.01	1.0	0.05	0.005	2.5
PCB 57	0.040	0.01	1.0	0.05	0.005	2.5
PCB 58	0.040	0.01	1.0	0.05	0.005	2.5
PCB 59	0.040	0.01	1.0	0.1	0.01	5
PCB 62	0.040	0.01	1.0	0.1	0.01	5
PCB 75	0.040	0.01	1.0	0.1	0.01	5
PCB 60	0.040	0.01	1.0	0.05	0.005	2.5
PCB 61	0.040	0.02	2.0	0.2	0.02	10
PCB 70	0.040	0.02	2.0	0.2	0.02	10
PCB 74	0.040	0.02	2.0	0.2	0.02	10
PCB 76	0.040	0.02	2.0	0.2	0.02	10
PCB 63	0.040	0.01	1.0	0.005	0.05	2.5
PCB 64	0.040	0.01	1.0	0.05	0.005	2.5
PCB 66	0.040	0.01	1.0	0.05	0.005	2.5
PCB 67	0.040	0.01	1.0	0.05	0.005	2.5
PCB 68	0.040	0.01	1.0	0.05	0.005	2.5
PCB 72	0.040	0.01	1.0	0.05	0.005	2.5
PCB 77	0.040	0.01	1.0	0.05	0.005	2.5
PCB 78	0.040	0.01	1.0	0.05	0.005	2.5
PCB 79	0.040	0.01	1.0	0.05	0.005	2.5
PCB 80	0.040	0.01	1.0	0.05	0.005	2.5
PCB 81	0.040	0.01	1.0	0.05	0.005	2.5
PCB 82	0.040	0.01	1.0	0.05	0.005	2.5
PCB 83	0.040	0.01	1.0	0.1	0.01	5
PCB 84	0.040	0.01	1.0	0.02	0.002	1
PCB 85	0.040	0.01	1.0	0.1	0.01	5
PCB 116	0.040	0.01	1.0	0.1	0.01	5
PCB 117	0.040	0.01	1.0	0.1	0.01	5
PCB 86	0.040	0.01	1.0	0.2	0.02	10
PCB 87	0.040	0.01	1.0	0.2	0.02	10
PCB 97	0.040	0.01	1.0	0.2	0.02	10
PCB 109	0.040	0.01	1.0	0.2	0.02	10
PCB 119	0.040	0.01	1.0	0.2	0.02	10
PCB 125	0.040	0.01	1.0	0.2	0.02	10
PCB 88	0.040	0.01	1.0	0.05	0.005	2.5
PCB 91	0.040	0.01	1.0	0.05	0.005	2.5
PCB 89	0.040	0.01	1.0	0.05	0.005	2.5
PCB 90	0.040	0.01	1.0	0.2	0.02	10
PCB 101	0.040	0.01	1.0	0.2	0.02	10
PCB 113	0.040	0.01	1.0	0.2	0.02	10
PCB 92	0.040	0.01	1.0	0.05	0.005	2.5
PCB 93	0.040	0.01	1.0	0.2	0.02	10
PCB 100	0.040	0.01	1.0	0.2	0.02	10
PCB 94	0.040	0.01	1.0	0.05	0.005	2.5
PCB 95	0.040	0.01	1.0	0.2	0.02	10
PCB 96	0.040	0.01	1.0	0.05	0.005	2.5
PCB 98	0.040	0.01	1.0	0.2	0.02	10
PCB 102	0.040	0.01	1.0	0.2	0.02	10
PCB 99	0.040	0.01	1.0	0.1	0.01	5
PCB 112	0.040	0.01	1.0	0.05	0.005	2.5
PCB 103	0.040	0.01	1.0	0.05	0.005	2.5
PCB 104	0.040	0.01	1.0	0.05	0.005	2.5
PCB 105	0.040	0.01	1.0	0.05	0.005	2.5
PCB 106	0.040	0.01	1.0	0.05	0.005	2.5
PCB 107	0.040	0.01	1.0	0.05	0.005	2.5
PCB 108	0.040	0.01	1.0	0.1	0.01	5
PCB 124	0.040	0.01	1.0	0.1	0.01	5
PCB 110	0.040	0.01	1.0	0.1	0.01	5

	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
Parameter	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**
PCB 115	0.040	0.01	1.0	0.1	0.01	5
PCB 111	0.040	0.01	1.0	0.05	0.005	2.5
PCB 114	0.040	0.01	1.0	0.05	0.005	2.5
PCB 118	0.040	0.01	1.0	0.1	0.01	5
PCB 120	0.040	0.01	1.0	0.05	0.005	2.5
PCB 121	0.040	0.01	1.0	0.05	0.005	2.5
PCB 122	0.040	0.01	1.0	0.05	0.005	2.5
PCB 123	0.040	0.01	1.0	0.05	0.005	2.5
PCB 126	0.040	0.01	1.0	0.05	0.005	2.5
PCB 127	0.040	0.01	1.0	0.05	0.005	2.5
PCB 128	0.040	0.01	1.0	0.1	0.01	5
PCB 166	0.040	0.01	1.0	0.1	0.01	5
PCB 129	0.040	0.01	1.0	0.2	0.02	10
PCB 138	0.040	0.01	1.0	0.2	0.02	10
PCB 163	0.040	0.01	1.0	0.2	0.02	10
PCB 130	0.040	0.01	1.0	0.05	0.005	2.5
PCB 131	0.040	0.01	1.0	0.05	0.005	2.5
PCB 132	0.040	0.01	1.0	0.05	0.005	2.5
PCB 133	0.040	0.01	1.0	0.05	0.005	2.5
PCB 134	0.040	0.01	1.0	0.1	0.01	5
PCB 143	0.040	0.01	1.0	0.1	0.01	5
PCB 135	0.040	0.01	1.0	0.1	0.01	5
PCB 151	0.040	0.01	1.0	0.1	0.01	5
PCB 136	0.040	0.01	1.0	0.05	0.005	2.5
PCB 137	0.040	0.01	1.0	0.05	0.005	2.5
PCB 164	0.040	0.01	1.0	0.05	0.005	2.5
PCB 139	0.040	0.01	1.0	0.1	0.01	5
PCB 140	0.040	0.01	1.0	0.1	0.01	5
PCB 141	0.040	0.01	1.0	0.05	0.005	2.5
PCB 142	0.040	0.01	1.0	0.05	0.005	2.5
PCB 144	0.040	0.01	1.0	0.05	0.005	2.5
PCB 145	0.040	0.01	1.0	0.05	0.005	2.5
PCB 146	0.040	0.01	1.0	0.05	0.005	2.5
PCB 147	0.040	0.01	1.0	0.1	0.01	5
PCB 149	0.040	0.01	1.0	0.1	0.01	5
PCB 148	0.040	0.01	1.0	0.05	0.005	2.5
PCB 150	0.040	0.01	1.0	0.05	0.005	2.5
PCB 152	0.040	0.01	1.0	0.05	0.005	2.5
PCB 153	0.040	0.01	1.0	0.1	0.01	5
PCB 168	0.040	0.01	1.0	0.1	0.01	5
PCB 154	0.040	0.01	1.0	0.1	0.01	5
PCB 155	0.040	0.01	1.0	0.05	0.005	2.5
PCB 156	0.040	0.01	1.0	0.1	0.01	5
PCB 157	0.040	0.01	1.0	0.1	0.01	5
PCB 158	0.040	0.01	1.0	0.05	0.005	2.5
PCB 159	0.040	0.01	1.0	0.05	0.005	2.5
PCB 160	0.040	0.01	1.0	0.2	0.02	10
PCB 161	0.040	0.01	1.0	0.05	0.005	2.5
PCB 162	0.040	0.01	1.0	0.05	0.005	2.5
PCB 165	0.040	0.01	1.0	0.05	0.005	2.5
PCB 167	0.040	0.01	1.0	0.05	0.005	2.5
PCB 169	0.040	0.01	1.0	0.05	0.005	2.5
PCB 170	0.040	0.01	1.0	0.05	0.005	2.5
PCB 171	0.040	0.01	1.0	0.1	0.01	5
PCB 173	0.040	0.01	1.0	0.1	0.01	5
PCB 172	0.040	0.01	1.0	0.05	0.005	2.5
PCB 174	0.040	0.01	1.0	0.05	0.005	2.5
PCB 175	0.040	0.01	1.0	0.05	0.005	2.5
PCB 176	0.040	0.01	1.0	0.05	0.005	2.5
PCB 177	0.040	0.01	1.0	0.05	0.005	2.5

	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
Parameter	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**
PCB 178	0.040	0.01	1.0	0.05	0.005	2.5
PCB 179	0.040	0.01	1.0	0.05	0.005	2.5
PCB 180	0.040	0.01	1.0	0.1	0.01	5
PCB 193	0.040	0.01	1.0	0.1	0.01	5
PCB 181	0.040	0.01	1.0	0.05	0.005	2.5
PCB 182	0.040	0.01	1.0	0.05	0.005	2.5
PCB 183	0.040	0.01	1.0	0.1	0.01	5
PCB 185	0.040	0.01	1.0	0.1	0.01	5
PCB 184	0.040	0.01	1.0	0.05	0.005	2.5
PCB 186	0.040	0.01	1.0	0.05	0.005	2.5
PCB 187	0.040	0.01	1.0	0.05	0.005	2.5
PCB 188	0.040	0.01	1.0	0.05	0.005	2.5
PCB 189	0.040	0.01	1.0	0.05	0.005	2.5
PCB 190	0.040	0.01	1.0	0.05	0.005	2.5
PCB 191	0.040	0.01	1.0	0.05	0.005	2.5
PCB 192	0.040	0.01	1.0	0.05	0.005	2.5
PCB 194	0.040	0.01	1.0	0.05	0.005	2.5
PCB 195	0.040	0.01	1.0	0.05	0.005	2.5
PCB 196	0.040	0.01	1.0	0.05	0.005	2.5
PCB 197	0.040	0.01	1.0	0.1	0.01	5
PCB 200	0.040	0.01	1.0	0.1	0.01	5
PCB 198	0.040	0.01	1.0	0.1	0.01	5
PCB 199	0.040	0.01	1.0	0.1	0.01	5
PCB 201	0.040	0.01	1.0	0.05	0.005	2.5
PCB 202	0.040	0.01	1.0	0.1	0.01	5
PCB 203	0.040	0.01	1.0	0.05	0.005	2.5
PCB 204	0.040	0.01	1.0	0.05	0.005	2.5
PCB 205	0.040	0.01	1.0	0.05	0.005	2.5
PCB 206	0.040	0.01	1.0	0.05	0.005	2.5
PCB 207	0.040	0.01	1.0	0.05	0.005	2.5
PCB 208	0.040	0.01	1.0	0.05	0.005	2.5
PCB 209	0.040	0.01	1.0	0.05	0.005	2.5

[1] The estimated minimum level (EML) is defined as the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present assuming a sample is extracted at the recommended weight or volume and is carried through all normal extraction and analysis procedures The values for other matrices (solids, tissues and solid wastes) reflect the EMLs for Protocol 1 as described in Table 12. If the sample is prepared by another protocol described in that section, the EMLs shown in this table must be adjusted appropriately. The EMLs are based on the mean plus 2 standard deviations for matrix-pooled historical blank data and calibration data obtained while performing EPA 1668A. The survey period was fourteen months, ending in February 2004. Individual EMLs may be adjusted to reflect more recent data.

[2] For reference purposes only, not currently used by TestAmerica Knoxville

[3] Other matrices include solids, tissues and solid wastes

Table 5a- Concentration of Native PCB Congener Stock and Spiking Solutions

Native PCB Congener	PCB	Standard Source	Catalog Number	Vendor Conc (ng/mL)	LCS Spiking Solution Conc (ng/mL)
2-MoCB	1	AccuStd	S-99994-4x	4000	5.0
4-MoCB	3	AccuStd	S-99994-4x	4000	5.0
2,2’-DiCB	4	AccuStd	S-99994-4x	4000	5.0
4,4’-DiCB	15	AccuStd	S-99994-4x	4000	5.0
2,2’,6-TrCB	19	AccuStd	S-99994-4x	4000	5.0
3,4,4’-TrCB	37	AccuStd	S-99994-4x	4000	5.0
2,2’,6,6’-TeCB	54	AccuStd	S-99994-4x	4000	5.0
3,3’,4,4’-TeCB	77	AccuStd	S-99994-4x	4000	5.0
3,4,4’,5-TeCB	81	AccuStd	S-99994-4x	4000	5.0
2,2’,4,6,6’-PeCB	104	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’-PeCB	105	AccuStd	S-99994-4x	4000	5.0
2,3,4,4’,5-PeCB	114	AccuStd	S-99994-4x	4000	5.0
2,3’,4,4’,5-PeCB	118	AccuStd	S-99994-4x	4000	5.0
2’,3,4,4’,5-PeCB	123	AccuStd	S-99994-4x	4000	5.0
3,3’,4,4’,5-PeCB	126	AccuStd	S-99994-4x	4000	5.0
2,2’,4,4’,6,6’-HxCB	155	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’,5-HxCB	156	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’,5’-HxCB	157	AccuStd	S-99994-4x	4000	5.0
2,3’,4,4’,5,5’-HxCB	167	AccuStd	S-99994-4x	4000	5.0
3,3’,4,4’,5,5’-HxCB	169	AccuStd	S-99994-4x	4000	5.0
2,2’,3,3’,4,4’,5-HpCB	170	AccuStd	S-99994-4x	4000	5.0
2,2’,3,4,4’,5,5’-HpCB	180	AccuStd	S-99994-4x	4000	5.0
2,2’,3,4’,5,6,6’-HpCB	188	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’,5,5’-HpCB	189	AccuStd	S-99994-4x	4000	5.0
2,2’,3,3’,5,5’,6,6’-OcCB	202	AccuStd	S-99994-4x	4000	5.0
2,3,3’,4,4’,5,5’,6-OcCB	205	AccuStd	S-99994-4x	4000	5.0
2,2’,3,3’,4,4’,5,5’,6-NoCB	206	AccuStd	S-99994-4x	4000	5.0
2,2’,3,3’,4’,5,5’,6,6’-NoCB	208	AccuStd	S-99994-4x	4000	5.0
DeCB	209	AccuStd	S-99994-4x	4000	5.0
All other CB congeners	NA	AccuStd	S-99994-4x	4000	5.0

Table 5b: Concentration of $^{13}C_{12}$ Labeled PCB Congener Stock and Spiking Solutions

Labeled PCB Congener	**PCB**	**Standard Source**	**Catalog Number**	**Vendor Conc (ng/mL)**	Stock Conc (ng/mL)	Spiking Solution Conc (ng/mL)
Internal Standards						
$^{13}C_{12}$-2-chlorobiphenyl	1L	Cambridge	EC-4908	40,000	1000	10
$^{13}C_{12}$-4-chlorobiphenyl	3L	Cambridge	EC-4990	40,000	1000	10
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	Cambridge	EC-4911	40,000	1000	10
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	Cambridge	EC-1402	40,000	1000	10
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	Cambridge	EC-4909	40,000	1000	10
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	Cambridge	EC-4901	40,000	1000	10
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	Cambridge	EC-4912	40,000	1000	10
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	Cambridge	EC-1404	40,000	1000	10
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	Cambridge	EC-1412	40,000	1000	10
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	Cambridge	EC-4910	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	Cambridge	EC-1420	40,000	1000	10
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	Cambridge	EC-4902	40,000	1000	10
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	Cambridge	EC-1435	40,000	1000	10
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	Cambridge	EC-4904	40,000	1000	10
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	Cambridge	EC-1425	40,000	1000	10
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	Cambridge	EC-4167	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	Cambridge	EC-1422	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	Cambridge	EC-4051	40,000	1000	10
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	Cambridge	EC-4050	40,000	1000	10
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	Cambridge	EC-1416	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	Cambridge	EC-4905	40,000	1000	10
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	Cambridge	EC-4913	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	Cambridge	EC-1409	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	Cambridge	EC-1408	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	Cambridge	EC-4199	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	Cambridge	EC-4900	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	Cambridge	EC-1419	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	Cambridge	EC-1410	40,000	1000	10
Recovery Standards						
$^{13}C_{12}$-2,5-dichlorobiphenyl	9L	Cambridge	EC-4165	40,000	1000	100
$^{13}C_{12}$-2,4',5-trichlorobiphenyl	31L	Wellington	MBP-31	50,000	1000	100
$^{13}C_{12}$-2,4',6-trichlorobiphenyl	32L	Cambridge	EC-4163	40,000	1000	100
$^{13}C_{12}$-2,2',5,5'-tetrachlorobiphenyl	52L	Cambridge	EC-1424	40,000	1000	100
$^{13}C_{12}$-2,2',4,5,5'-pentachlorobiphenyl	101L	Cambridge	EC-1405	40,000	1000	100
$^{13}C_{12}$-3,3',4,5,5'-pentachlorobiphenyl	127L	Cambridge	EC-1421	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5'-hexachlorobiphenyl	138L	Cambridge	EC-1436	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-heptachlorobiphenyl	180L	Cambridge	EC-1407	40,000	1000	100
$^{13}C_{12}$-2,2',3,3',4,4’,5,5'-octachlorobiphenyl	194L	Cambridge	EC-1418	40,000	1000	100
Cleanup Standards						
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	Cambridge	EC-1413	40,000	5000	10
$^{13}C_{12}$-2,3,3’,5,5’-pentachlorobiphenyl	111L	Cambridge	EC-1415	40,000	5000	10
$^{13}C_{12}$-2,2’,3,3’,5,5’,6-heptachlorobiphenyl	178L	Cambridge	EC-1417	40,000	5000	10
Sampling Surrogate Standards						
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	Cambridge	EC-5095	40,000	5000	50
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	Cambridge	EC-5048	40,000	5000	50
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	Wellington	MBP-95	50,000	5000	50
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	Cambridge	EC-1406	40,000	5000	50

Table 6a - Concentration of PCBs in Calibration Solutions

Analyte Type	PCB[1]	CS 0.5 ng/mL	CS 1 ng/mL	CS 2 ng/mL	CS 3[2] ng/mL	CS 4 ng/mL	CS 5 ng/mL
Congeners							
2-MoCB	1	0.5	1.0	5.0	50	400	2000
4-MoCB	3	0.5	1.0	5.0	50	400	2000
2,2'-DiCB	4	0.5	1.0	5.0	50	400	2000
4,4'-DiCB	15	0.5	1.0	5.0	50	400	2000
2,2',6'-TrCB	19	0.5	1.0	5.0	50	400	2000
3,4,4'-TrCB	37	0.5	1.0	5.0	50	400	2000
2,2',6,6'-TeCB	54	0.5	1.0	5.0	50	400	2000
3,3',4,4'-TeCB	77	0.5	1.0	5.0	50	400	2000
3,4,4',5-TeCB	81	0.5	1.0	5.0	50	400	2000
2,2',4,6,6'-PeCB	104	0.5	1.0	5.0	50	400	2000
2,3,3',4,4'-PeCB	105	0.5	1.0	5.0	50	400	2000
2,3,4,4',5-PeCB	114	0.5	1.0	5.0	50	400	2000
2,3',4,4',5-PeCB	118	0.5	1.0	5.0	50	400	2000
2',3,4,4',5-PeCB	123	0.5	1.0	5.0	50	400	2000
3,3',4,4',5-PeCB	126	0.5	1.0	5.0	50	400	2000
2,2',4,4',6,6'-HxCB	155	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5-HxCB	156	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5'-HxCB	157	0.5	1.0	5.0	50	400	2000
2,3',4,4',5,5'-HxCB	167	0.5	1.0	5.0	50	400	2000
3,3',4,4',5,5'-HxCB	169	0.5	1.0	5.0	50	400	2000
2,2',3,4',5,6,6'-HpCB	188	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5,5'-HpCB	189	0.5	1.0	5.0	50	400	2000
2,2',3,3',5,5',6,6'-OcCB	202	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5,5',6-OcCB	205	0.5	1.0	5.0	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB	206	0.5	1.0	5.0	50	400	2000
2,2',3,3',4',5,5',6,6'-NoCB	208	0.5	1.0	5.0	50	400	2000
DeCB	209	0.5	1.0	5.0	50	400	2000
All other CB congeners		0.5	1.0	5.0	50	400	2000
Labeled Congeners							
$^{13}C_{12}$-2-MoCB	1L	100	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	3L	100	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	4L	100	100	100	100	100	100
$^{13}C_{12}$-4,4'-DiCB	15L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6-TrCB	19L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4’,5-TeCB	81L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100

Analyte Type	PCB[1]	CS 0.5 ng/mL	CS 1 ng/mL	CS 2 ng/mL	CS 3[2] ng/mL	CS 4 ng/mL	CS 5 ng/mL
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4',5,5',6,6'-NoCB	208L	100	100	100	100	100	100
$^{13}C_{12}$-DeCB	209L	100	100	100	100	100	100
Cleanup Standards							
$^{13}C_{12}$-2,4,4'-TriCB	28L			5.0	50	400	--
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L			5.0	50	400	--
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L			5.0	50	400	--
Recovery Standards							
$^{13}C_{12}$-2,5-DiCB	9L	100	100	100	100	100	100
$^{13}C_{12}$-2,4',5-TriCB	31L	100	100	100	100	100	100
$^{13}C_{12}$-2,4',6-TriCB	32L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,5,5'-PeCB	127L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100
Labeled Sampling Surrogates							
$^{13}C_{12}$-2,4'-DiCB	8L			5.0	50	400	--
$^{13}C_{12}$-3,3',4,5'-TeCB	79L			5.0	50	400	--
$^{13}C_{12}$-2,2',3,5',6-PeCB	95L			5.0	50	400	--
$^{13}C_{12}$-2,2',4,4',5,5'-HxCB	153L			5.0	50	400	--

Notes:

1. Suffix “L” indicates labeled compound.

2. The CS 3 standard is also used as the calibration verification solution.

Table 6b – Preparation of Calibration Solutions

Calibration Standard ID	Parent Standards	Parent Conc. (ng/mL)	Volume Added (mL)	Final Volume (mL)	Final Conc (ng/mL)
CS 0.5	Native PCB Congener Stock Solution	40	0.0125	1.0	0.50
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100
CS 1	Native PCB Congener Stock Solution	40	0.0250	1.0	1.0
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100
CS 2	Native PCB Congener Stock Solution	40	0.1250	1.0	5.0
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100
	Cleanup Standard Stock Solution	5000	0.0010		5.0
	Sampling Surrogate Stock Solution	5000	0.0010		5.0
CS 3	Native PCB Congener Standard Mix	4000	0.0375	3.0	50
	Internal Standard Stock Solution	1000	0.3000		100
	Recovery Standard Stock Solution	1000	0.3000		100
	Cleanup Standard Stock Solution	5000	0.0300		50
	Sampling Surrogate Stock Solution	5000	0.0300		50
CS 4	Native PCB Congener Standard Mix	4000	0.1000	1.0	400
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100
	Cleanup Standard Stock Solution	5000	0.0800		400
	Sampling Surrogate Stock Solution	5000	0.0800		400
CS 5	Native PCB Congener Standard Mix	4000	0.5000	1.0	2000
	Internal Standard Stock Solution	1000	0.1000		100
	Recovery Standard Stock Solution	1000	0.1000		100

Table 7 - GC Window Defining Mixture and SPB –Octyl Resolution Test Compounds

SPB-Octyl

Congener Group	First Eluted		Last Eluted	
Mono	1	2-	3	4-
Di	4	2,2’-	15	4,4’-
Tri	19	2,2’,6-	37	3,4,4’-
Tetra	54	2,2’,6,6’-	77	3,3’,4,4’-
Penta	104	2,2’,4,6,6’-	126	3,3’,4,4’,5-
Hexa	155	2,2’,4,4’,6,6’-	169	3,3’,4,4’,5,5’-
Hepta	188	2,2’,3,4’,5,6,6’-	189	2,3,3’,4,4’,5,5’-
Octa	202	2,2’,3,3’,5,5’,6,6’-	205	2,3,3’,4,4’,5,5’,6-
Nona	208	2,2’,3,3’,4,5,5’,6,6’-	206	2,2’,3,3’,4,4’,5,5’,6-
Deca	209	2,2’,3,3’,4,4’,5,5’,6,6’-	209	2,2’,3,3’,4,4’,5,5’,6,6’-

SPB-Octyl Resolution Test Compounds

23	2,3,5-trichlorobiphenyl
34	2’,3,5-trichlorobiphenyl (2,3’,5’-trichlorobiphenyl)
182	2,2’,3,4,4’,5,6’-heptachlorobiphenyl
187	2,2’,3,4’,5,5’,6-heptachlorobiphenyl

Table 8 - Ions Monitored for HRGC/HRMS Analysis of PCBs

Descriptor	Accurate Mass	Ion ID	Elemental Composition	Analyte
1	180.9888	Lock	C_4F_7	PFK
	188.0393	M	$C_{12}H_9{}^{35}Cl$	Mono
	190.0363	M+2	$C_{12}H_9{}^{37}Cl$	Mono
	200.0795	M	$^{13}C_{12}H_9{}^{35}Cl$	Mono-13C12
	202.0766	M+2	$^{13}C_{12}H_9{}^{37}Cl$	Mono-13C12
	222.0003	M	$C_{12}H_8{}^{35}Cl_2$	Di
	223.9974	M+2	$C_{12}H_8{}^{35}Cl^{37}Cl$	Di
	234.0406	M	$^{13}C_{12}H_8{}^{35}Cl_2$	Di-13C
	236.0376	M+2	$^{13}C_{12}H_8{}^{35}Cl^{37}Cl$	Di-13C
	255.9613	M	$C_{12}H_7{}^{35}Cl_3$	Tri
	257.9584	M+2	$C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	Tri-13C
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri-13C
	280.9824	QC	C_6F_{11}	PFK
	289.9224	M	$C_{12}H_6{}^{35}Cl_4$	Tetra
	291.9194	M+2	$C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_4$	Tetra-13C
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra-13C
2	255.9613	M	$C_{12}H_7{}^{35}Cl_3$	Tri
	257.9584	M+2	$C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	Tri-13C
	268.9824	Lock	C_5F_{11}	PFK
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri-13C
	289.9224	M	$C_{12}H_6{}^{35}Cl_4$	Tetra
	291.9194	M+2	$C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_4$	Tetra-13C
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra-13C
	325.8804	M+2	$C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta
	327.8775	M+4	$C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta-13C
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta-13C
	359.8415	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa
	361.8385	M+4	$C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa-13C
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa-13C
	380.9760	QC	$C_{10}F_{14}$	PFK

Table 8 - Ions Monitored for HRGC/HRMS Analysis of PCBs (continued)

Descriptor	Accurate Mass	Ion ID	Elemental Composition	Analyte
3	325.8804	M+2	$C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta
	327.8775	M+4	$C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta-13C
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta-13C
	342.9792	Lock	C_8F_{13}	PFK
	359.8415	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa
	361.8385	M+4	$C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa-13C
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa-13C
	393.8025	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta
	395.7995	M+4	$C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta-13C
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta-13C
	427.7635	M+2	$C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa
	429.7606	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa
	430.9728	QC	C_9F_{17}	PFK
	439.8038	M+2	$^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa-13C
	441.8008	M+4	$^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa-13C
4	393.8025	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta
	395.7995	M+4	$C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta
	404.9760	Lock	$C_{10}F_{15}$	PFK
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta-13C
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta-13C
	427.7635	M+2	$C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa
	429.7606	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa
	439.8038	M+2	$^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa-13C
	441.8008	M+4	$^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa-13C
	461.7246	M+2	$C_{12}H^{35}Cl_7{}^{37}Cl_2$	Nona
	463.7216	M+4	$C_{12}H^{35}Cl_6{}^{37}Cl_3$	Nona
	473.7648	M+2	$^{13}C_{12}H^{35}Cl_7{}^{37}Cl_2$	Nona-13C
	475.7619	M+4	$^{13}C_{12}H^{35}Cl_6{}^{37}Cl_3$	Nona-13C
	495.6856	M+2	$C_{12}{}^{35}Cl_8{}^{37}Cl_2$	Deca
	497.6826	M+4	$C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Deca
	504.9697	QC	$C_{12}F_{19}$	PFK
	507.7258	M+2	$^{13}C_{12}{}^{35}Cl_8{}^{37}Cl_2$	Deca-13C
	509.7229	M+4	$^{13}C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Deca-13C

1. Nuclidic masses used: H = 1.007825 C = 12.00000 ^{13}C = 13.003355 F = 18.9984

O = 15.994915 ^{35}Cl = 34.968853 ^{37}Cl = 36.965903

Table 9 - Theoretical Ion Abundance Ratios and Control Limits for PCBs

Chlorine Atoms	mass's Forming Ratios	Theoretical Ratio	Lower QC Limit	Upper QC Limit
1	m/m+2	3.13	2.66	3.60
2	m/m+2	1.56	1.33	1.79
3	m/m+2	1.04	0.88	1.20
4	m/m+2	0.77	0.65	0.89
5	m+2/m+4	1.55	1.32	1.78
6	m+2/m+4	1.24	1.05	1.43
7	m+2/m+4	1.05	0.89	1.21
8	m+2/m+4	0.89	0.76	1.02
9	m+2/m+4	0.77	0.65	0.89
10	m+2/m+4	0.69	0.59	0.79

Table 10A - Acceptance Criteria for Performance Tests (1668A & B)

			IPR (IDOC)			
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL)** [1]	**%RSD**	**%R**	**VER %R**	**LCS %R**
2-chlorobiphenyl	1	50	40	60-140	70-130	50-150
4-chlorobiphenyl	3	50	40	60-140	70-130	50-150
2,2'-dichlorobiphenyl	4	50	40	60-140	70-130	50-150
4,4'-dichlorobiphenyl	15	50	40	60-140	70-130	50-150
2,2',6-trichlorobiphenyl	19	50	40	60-140	70-130	50-150
3,4,4'-trichlorobiphenyl	37	50	40	60-140	70-130	50-150
2,2',6,6'-tetrachlorobiphenyl	54	50	40	60-140	70-130	50-150
3,3',4,4'-tetrachlorobiphenyl	77	50	40	60-140	70-130	50-150
3,4,4',5-tetrachlorobiphenyl	81	50	40	60-140	70-130	50-150
2,2'4,6,6'-pentachlorobiphenyl	104	50	40	60-140	70-130	50-150
2,3,3',4,4'-pentachlorobiphenyl	105	50	40	60-140	70-130	50-150
2,3,4,4',5-pentachlorobiphenyl	114	50	40	60-140	70-130	50-150
2,3',4,4',5-pentachlorobiphenyl	118	50	40	60-140	70-130	50-150
2',3',4,4',5-pentachlorobiphenyl	123	50	40	60-140	70-130	50-150
3,3',4,4',5-pentachlorobiphenyl	126	50	40	60-140	70-130	50-150
2,2',4,4',6,6'-hexachlorobiphenyl	155	50	40	60-140	70-130	50-150
2,3,3',4,4',5-hexachlorobiphenyl	156	50	40	60-140	70-130	50-150
2,3',4,4',5,5'-hexachlorobiphenyl	157	50	40	60-140	70-130	50-150
2,3,3',4,4',5'-hexachlorobiphenyl	167	50	40	60-140	70-130	50-150
3,3',4,4',5,5'-hexachlorobiphenyl	169	50	40	60-140	70-130	50-150
2,2',3,4'5,6,6'-heptachlorobiphenyl	188	50	40	60-140	70-130	50-150
2,3,3',4,4',5,5'-heptachlorobiphenyl	189	50	40	60-140	70-130	50-150
2,2',3,3',5,5',6,6'-octachlorobiphenyl	202	50	40	60-140	70-130	50-150
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	50	40	60-140	70-130	50-150
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206	50	40	60-140	70-130	50-150
2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208	50	40	60-140	70-130	50-150
2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209	50	40	60-140	70-130	50-150
Internal Standards						
$^{13}C_{12}$-2-chlorobiphenyl	1L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-4-chlorobiphenyl	3L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	100	50	35-135	50-150	30-140
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	100	50	35-135	50-150	30-140

			IPR (IDOC)			
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL)** [1]	**%RSD**	**%R**	**VER %R**	**LCS %R**
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	100	50	35-135	50-150	30-140
Cleanup Standards						
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	50	45	45-120	60-130	40-125
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	50	45	45-120	60-130	40-125
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	50	45	45-120	60-130	40-125
Sampling Surrogate Standards						
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	50			50-150	50-150
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	50			50-150	50-150
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	50			50-150	50-150
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	50			50-150	50-150

[1] - Test concentrations are based on ng/mL in the sample extract or standard solution.

Table 10B - Acceptance Criteria for Performance Tests (1668C)

			IPR (IDOC)				
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL)** [1]	**%RSD**	**Mean %R**	**VER %R**	**OPR (LCS) %R**	**Labeled Compound %R in Samples**
2-chlorobiphenyl	1	50	25	70-130	75-125	60-135	
4-chlorobiphenyl	3	50	25	70-130	75-125	60-135	
2,2’-dichlorobiphenyl	4	50	25	70-130	75-125	60-135	
4,4’-dichlorobiphenyl	15	50	25	70-130	75-125	60-135	
2,2’,6-trichlorobiphenyl	19	50	25	70-130	75-125	60-135	
3,4,4’-trichlorobiphenyl	37	50	25	70-130	75-125	60-135	
2,2’,6,6’-tetrachlorobiphenyl	54	50	25	70-130	75-125	60-135	
3,3’,4,4’-tetrachlorobiphenyl	77	50	25	70-130	75-125	60-135	
3,4,4’,5-tetrachlorobiphenyl	81	50	25	70-130	75-125	60-135	
2,2’4,6,6’-pentachlorobiphenyl	104	50	25	70-130	75-125	60-135	
2,3,3’,4,4’-pentachlorobiphenyl	105	50	25	70-130	75-125	60-135	
2,3,4,4’,5-pentachlorobiphenyl	114	50	25	70-130	75-125	60-135	
2,3’,4,4’,5-pentachlorobiphenyl	118	50	25	70-130	75-125	60-135	
2’,3’,4,4’,5-pentachlorobiphenyl	123	50	25	70-130	75-125	60-135	
3,3’,4,4’,5-pentachlorobiphenyl	126	50	25	70-130	75-125	60-135	
2,2’,4,4’,6,6’-hexachlorobiphenyl	155	50	25	70-130	75-125	60-135	
2,3,3’,4,4’,5-hexachlorobiphenyl	156	50	25	70-130	75-125	60-135	
2,3’,4,4’,5,5’-hexachlorobiphenyl	157	50	25	70-130	75-125	60-135	
2,3,3’,4,4’,5’-hexachlorobiphenyl	167	50	25	70-130	75-125	60-135	
3,3’,4,4’,5,5’-hexachlorobiphenyl	169	50	25	70-130	75-125	60-135	
2,2’,3,4’5,6,6’-heptachlorobiphenyl	188	50	25	70-130	75-125	60-135	
2,3,3’,4,4’,5,5’-heptachlorobiphenyl	189	50	25	70-130	75-125	60-135	
2,2’,3,3',5,5',6,6’-octachlorobiphenyl	202	50	25	70-130	75-125	60-135	
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	50	25	70-130	75-125	60-135	
2,2’,3,3’,4,4’,5,5’,6-nonachlorobiphenyl	206	50	25	70-130	75-125	60-135	
2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl	208	50	25	70-130	75-125	60-135	
2,2’,3,3’,4,4’,5,5’,6,6’-decachlorobiphenyl	209	50	25	70-130	75-125	60-135	
Internal Standards							
$^{13}C_{12}$-2-chlorobiphenyl	1L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-4-chlorobiphenyl	3L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2’-dichlorobiphenyl	4L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-4,4’-dichlorobiphenyl	15L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2’,6-trichlorobiphenyl	19L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-3,4,4’-trichlorobiphenyl	37L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2’,6,6’-tetrachlorobiphenyl	54L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-3,3’,4,4’-tetrachlorobiphenyl	77L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,4,4’,5-tetrachlorobiphenyl	81L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,4,6,6’-pentachlorobiphenyl	104L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’-pentachlorobiphenyl	105L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$2,3,4,4’,5-pentachlorobiphenyl -	114L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3’,4,4’,5-pentachlorobiphenyl	118L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2’,3,4,4’,5-pentachlorobiphenyl	123L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,3’,4,4’,5-pentachlorobiphenyl	126L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,4,4’,6,6’-hexachlorobiphenyl	155L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’,5-hexachlorobiphenyl	156L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’,5’-hexachlorobiphenyl	157L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3’,4,4’,5,5’-hexachlorobiphenyl	167L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,3’,4,4’,5,5’-hexachlorobiphenyl	169L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,4’,5,6,6’-heptachlorobiphenyl	188L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3’,4,4’,5,5’-heptachlorobiphenyl	189L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,5,5’,6,6’-octachlorobiphenyl	202L	100	50	45-135	50-145	40-145	10-145

			IPR (IDOC)				
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL)** [1]	**%RSD**	**Mean %R**	**VER %R**	**OPR (LCS) %R**	**Labeled Compound %R in Samples**
$^{13}C_{12}$-2,3,3’,4,4’,5,5’,6-octachlorobiphenyl	205L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,4,4’,5,5’,6-nonachlorobiphenyl	206L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl	208L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,4,4’,5,5’,6,6’-decachlorobiphenyl	209L	100	50	45-135	50-145	40-145	10-145
Cleanup Standards							
$^{13}C_{12}$-2,4,4’-trichlorobiphenyl	28L	50	70	20-135	65-135	15-145	5-145
$^{13}C_{12}$-2,3,3’,5,5’-pentachlorobiphenyl	111L	50	50	45-135	75-125	40-145	10-145
$^{13}C_{12}$-2,2’,3,3’,5,5’,6-heptachlorobiphenyl	178L	50	50	45-135	75-125	40-145	10-145
Sampling Surrogate Standards							
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	50			50-150	50-150	50-150
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	50			50-150	50-150	50-150
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	50			50-150	50-150	50-150
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	50			50-150	50-150	50-150

[1] - Test concentrations are based on ng/mL in the sample extract or standard solution.

Table 11 - Retention Times of Isomers on the SPB-Octyl Column for the PCB Standard Mixes[1]

PCB Congener Mix 1 (AccustandardM-1668-1)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
1	2	4	78	6	161
2	10	4	81	6	153
2	9	5	96	6	130
2	6	5	103	6	129
2	8	5	95	6	166
2	14	5	88	6	159
2	11	5	89	6	167
3	30	5	92	6	156
3	27	5	113	7	179
3	32	5	83	7	176
3	34	5	119	7	178
3	26	5	87	7	175
3	31	5	85	7	183
3	33	5	82	7	177
3	36	5	120	7	171
3	38	5	124	7	172
3	35	5	106	7	191
4	50	5	122	7	170
4	45	5	105	7	190
4	52	5	127	8	200/201
4	49	6	152	8	204
4	75	6	136	8	199/200
4	41	6	148	8	198
4	72	6	151	8	196
4	57	6	144	8	195
4	63	6	143	8	194
4	66	6	142	9	207
4	79	6	133		

PCB Congener Mix 2 (Accustandard M-1668-2)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
2	7	4	55	6	139
2	5	4	60	6	132
2	12	5	94	6	165
3	18	5	100	6	168
3	24	5	91	6	137
3	23	5	121	6	160
3	28	5	90	6	128
3	22	5	99	6	162
3	39	5	109/108	6	157
4	53	5	117	7	184
4	51	5	111	7	186
4	73	5	108/107	7	187
4	48	5	118	7	185
4	62	5	114	7	181
4	71	6	150	7	192
4	68	6	145	8	197
4	58	6	135	8	201/199
4	61	6	149	8	203

Table 11 - Retention Times of Isomers on the SPB-Octyl Column for the PCB Standard Mixes[1] (Continued)

PCB Congener Mix 3 (Accustandard M-1668-3)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
2	13	4	80	6	140
3	17	5	93	6	146
3	29	5	84	6	141
3	20	5	101	6	164
4	46	5	112	6	158
4	65	5	86	7	182
4	59	5	116	7	174
4	40	5	107/109	7	173
4	67	6	154	7	193
4	76	6	147		

PCB Congener Mix 4 (Accustandard M-1668-4)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
3	25	4	64	5	123
3	21	4	70	6	134
4	69	5	102	6	131
4	47	5	97	6	163
4	42	5	115	7	180

PCB Congener Mix 5 (AccustandardM-1668-5)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
1	1	4	74	6	169
1	3	4	56	7	188
2	4	4	77	7	189
2	15	5	104	8	202
3	19	5	98	8	205
3	16	5	125	9	208
3	37	5	110	9	206
4	54	5	126	10	209
4	43	6	155		
4	44	6	138		

Notes:

1 Each congener mix is analyzed in triplicate to establish the retention times of the PCB isomers in the absence of co-eluting isomers. The elution order listed here is used to assign peak identifications in the separate mixture analysis. The average retention time established in the analysis of the separate mixtures is then used to establish relative retention times. (See sections 10.2.3)

2 BZ/IUPAC Number, if different.

Table 12: Assignment of Sample Preparation Protocols

Figure 1 - Recommended GC Operating Conditions

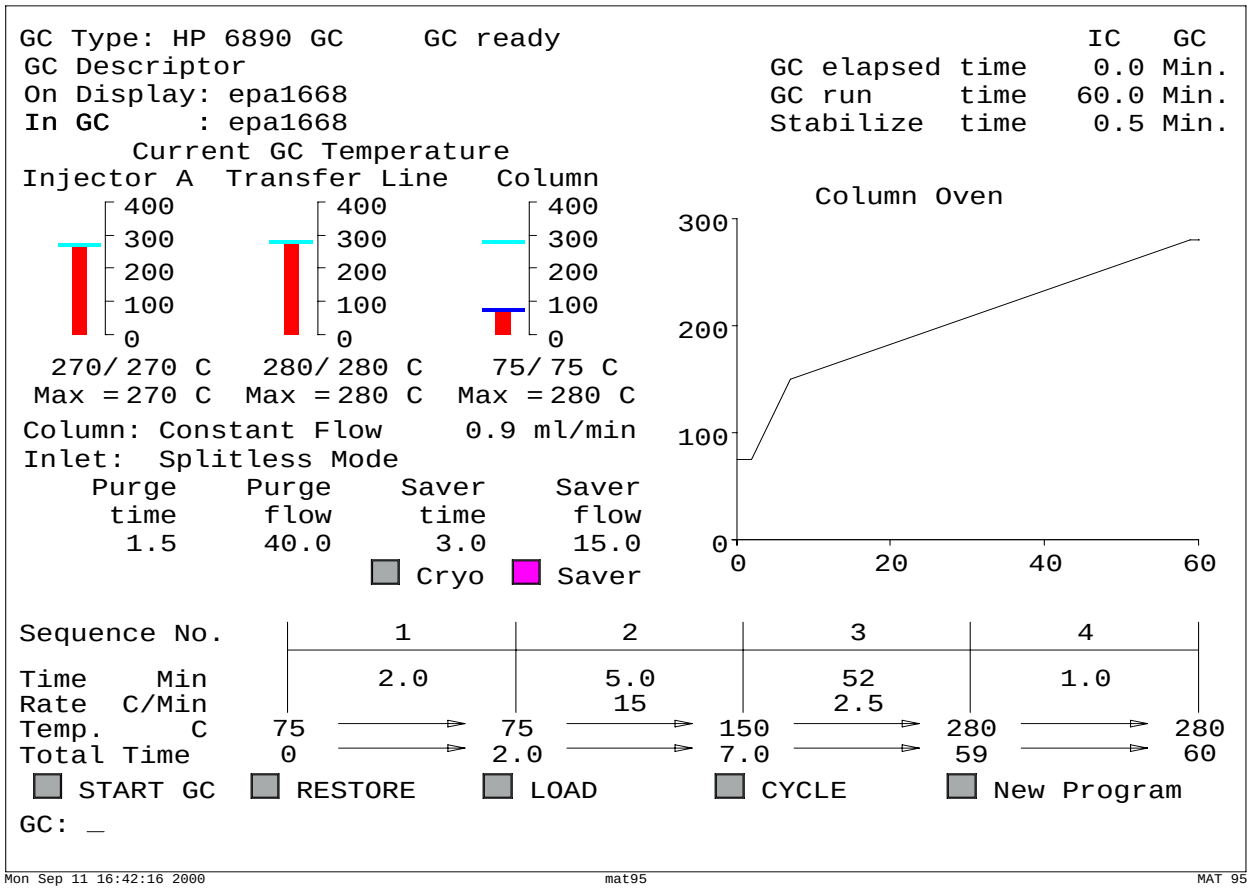

Figure 2 - Recommended MID Descriptors

```
MID Set Up Parameters
 MID File                    epa1668
 Measure/lock ratio (X)          1
 Set Damping relay  (T)     FALSE
 Width first lock   (A)      0.15 amu
 Electric jump time (E)        10 ms
 Magnetic jump time (D)        60 ms
 Offset             (O)       100 cts
 Electric range     (R)       300 %
 Sweep peak width   (W)      3.00
 Acq mode         (C|P)      Cent mode
 MID mode     (J|M|L|N)      Lock mode

 MID  Time Windows      >   <   ^   v
 #  Start  Measure  End      Cycletime
 1   8:00  15:45  23:45 min   1.00 sec
 2  23:45  14:15  38:00 min   1.00 sec
 3  38:00  13:15  51:15 min   1.00 sec
 4  51:15   8:45  60:00 min   1.00 sec
 5
 6
 7
 8
 9

 Clear Menu      Clear Times      Clear Masses
 Start MID       RESTORE          Main

MID: _
```

```
 MID Masses for Time Window   1
 #      mass    F int  gr time(ms)
 1    188.0393      1   1    47.79
 2    190.0363      1   1    47.79
 3    192.9888 l   10   1     4.10
 4    194.0594      1   1    47.79
 5    196.0565      1   1    47.79
 6    200.0795      1   1    47.79
 7    202.0766      1   1    47.79
 8    222.0003      1   1    47.79
 9    223.9974      1   1    47.79
10    234.0406      1   1    47.79
11    236.0376      1   1    47.79
12    255.9613      1   1    47.79
13    257.9584      1   1    47.79
14    268.0016      1   1    47.79
15    268.9824 c   10   1     4.10
16    269.9986      1   1    47.79
17    289.9224      1   1    47.79
18    291.9194      1   1    47.79
19
20
21
22
23
24
 >    Lock Mass    Cali Mass
```

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```
MID Set Up Parameters
 MID File                    epa1668
 Measure/lock ratio (X)          1
 Set Damping relay  (T)     FALSE
 Width first lock   (A)      0.15 amu
 Electric jump time (E)        10 ms
 Magnetic jump time (D)        60 ms
 Offset             (O)       100 cts
 Electric range     (R)       300 %
 Sweep peak width   (W)      3.00
 Acq mode         (C|P)      Cent mode
 MID mode     (J|M|L|N)      Lock mode

 MID  Time Windows      >   <   ^   v
 #  Start  Measure  End      Cycletime
 1   8:00  15:45  23:45 min   1.00 sec
 2  23:45  14:15  38:00 min   1.00 sec
 3  38:00  13:15  51:15 min   1.00 sec
 4  51:15   8:45  60:00 min   1.00 sec
 5
 6
 7
 8
 9

 Clear Menu      Clear Times      Clear Masses
 Start MID       RESTORE          Main

MID: _
```

```
 MID Masses for Time Window   2
 #      mass    F int  gr time(ms)
 1    255.9613      1   1    55.98
 2    257.9584      1   1    55.98
 3    268.0016      1   1    55.98
 4    268.9824 l   10   1     5.46
 5    269.9986      1   1    55.98
 6    289.9224      1   1    55.98
 7    291.9194      1   1    55.98
 8    301.9626      1   1    55.98
 9    303.9597      1   1    55.98
10    325.8804      1   1    55.98
11    327.8775      1   1    55.98
12    337.9207      1   1    55.98
13    339.9178      1   1    55.98
14    342.9792 c   10   1     5.46
15    359.8415      1   1    55.98
16    361.8385      1   1    55.98
17
18
19
20
21
22
23
24
 >    Lock Mass    Cali Mass
```

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Figure 2 - Recommended MID Descriptors (Continued)

```
MID Set Up Parameters
 MID File                    epa1668
 Measure/lock ratio (X)          1
 Set Damping relay  (T)     FALSE
 Width first lock   (A)      0.15 amu
 Electric jump time (E)        10 ms
 Magnetic jump time (D)        60 ms
 Offset             (O)       100 cts
 Electric range     (R)       300 %
 Sweep peak width   (W)      3.00
 Acq mode         (C|P)      Cent mode
 MID mode     (J|M|L|N)      Lock mode

 MID  Time Windows      >   <   ^   v
 #  Start  Measure  End      Cycletime
 1   8:00  15:45  23:45 min   1.00 sec
 2  23:45  14:15  38:00 min   1.00 sec
 3  38:00  13:15  51:15 min   1.00 sec
 4  51:15   8:45  60:00 min   1.00 sec
 5
 6
 7
 8
 9

 Clear Menu      Clear Times      Clear Masses
 Start MID       RESTORE          Main

MID: _
```

```
 MID Masses for Time Window   3
 #      mass    F int  gr time(ms)
 1    325.8804      1   1    47.79
 2    327.8775      1   1    47.79
 3    337.9207      1   1    47.79
 4    339.9178      1   1    47.79
 5    342.9792 l   10   1     4.10
 6    359.8415      1   1    47.79
 7    361.8385      1   1    47.79
 8    371.8817      1   1    47.79
 9    373.8788      1   1    47.79
10    393.8025      1   1    47.79
11    395.7995      1   1    47.79
12    405.8428      1   1    47.79
13    407.8398      1   1    47.79
14    427.7635      1   1    47.79
15    429.7606      1   1    47.79
16    430.9728 c   10   1     4.10
17    439.8038      1   1    47.79
18    441.8008      1   1    47.79
19
20
21
22
23
24
 >    Lock Mass    Cali Mass
```

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```
MID Set Up Parameters
 MID File                    epa1668
 Measure/lock ratio (X)          1
 Set Damping relay  (T)     FALSE
 Width first lock   (A)      0.15 amu
 Electric jump time (E)        10 ms
 Magnetic jump time (D)        60 ms
 Offset             (O)       100 cts
 Electric range     (R)       300 %
 Sweep peak width   (W)      3.00
 Acq mode         (C|P)      Cent mode
 MID mode     (J|M|L|N)      Lock mode

 MID  Time Windows      >   <   ^   v
 #  Start  Measure  End      Cycletime
 1   8:00  15:45  23:45 min   1.00 sec
 2  23:45  14:15  38:00 min   1.00 sec
 3  38:00  13:15  51:15 min   1.00 sec
 4  51:15   8:45  60:00 min   1.00 sec
 5
 6
 7
 8
 9

 Clear Menu      Clear Times      Clear Masses
 Start MID       RESTORE          Main

MID: _
```

```
 MID Masses for Time Window   4
 #      mass    F int  gr time(ms)
 1    393.8025      1   1    47.79
 2    395.7995      1   1    47.79
 3    404.9760 l   10   1     4.10
 4    405.8428      1   1    47.79
 5    407.8398      1   1    47.79
 6    427.7635      1   1    47.79
 7    429.7606      1   1    47.79
 8    439.8038      1   1    47.79
 9    441.8008      1   1    47.79
10    463.7216      1   1    47.79
11    465.7187      1   1    47.79
12    475.7619      1   1    47.79
13    477.7589      1   1    47.79
14    497.6826      1   1    47.79
15    499.6797      1   1    47.79
16    504.9697 c   10   1     4.10
17    509.7229      1   1    47.79
18    511.7199      1   1    47.79
19
20
21
22
23
24
 >    Lock Mass    Cali Mass
```

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Figure 3 - Example Data Review Checklist

TestAmerica Knoxville Specialty Organics Group GC/MS Initial Calibration Data Review Checklist
Method or SOP Number: KNOX-ID-0013 Revision 11

PFK Date/Time:		Inst:		2nd Source Filename:	

CS0.5 Filename	CS1 Filename	CS2 Filename	CS3 Filename	CS4 Filename	CS5 Filename

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd Level
1. Was the mass resolution documented before beginning the initial calibration?					
2. Was the instrument resolution ≥8,000 throughout (≥ 10,000 for m/z 342.9792) and ≥10,000 in the center of each m/z range. PFK m/z 192.9888, *230.9856, and *280.9824? PFK m/z 268.9824, *292.9824, and *380.9760? PFK m/z 342.9792, *380.9760, and *430.9728? PFK m/z 404.9760, *442.9728, and *530.9665?					
3. Were the measured exact masses listed above within 5 ppm at reduced accelerating voltage?					
4. Have PCB Mixes 1 - 5 been analyzed in triplicate using the installed column to assign congener retention times, method retention times, and MID switch points?					
5. Were the calibration standard solutions, at the number and concentrations specified in the Method/SOP, analyzed?					
6. Was date/time of analysis verified between analysis header and logbook as correct?					
7. Was the valley height less than 40% of the height of the shorter of the two peaks for the pair PCB 23 and PCB 34, and the pair PCB 182 and PCB 187 in the CS3 standard?					
8. Was the absolute retention time of PCB 209 greater than 55 minutes in the CS3 standard?					
9. Were the response factors calculated for each labeled standard and unlabeled native analyte using the Method/SOP specified reference compound, quantitation ions, and formula?					
10. Is the %RSD acceptable for all native analytes (within ± 20% calculated by isotope dilution, within ± 35% calculated by internal standard)?					
11. Is the %RSD acceptable (within ± 35%) for all labeled standards?					
12. Are all S/N ratios ≥10 for the GC signals in each EICP (extracted ion chromatographic profile) including internal standards (Exception: Secondary native dichloro biphenyl channel m/z 223.9974)?					
13. Are the ion abundance ratios for all native Toxics/LOCs and all labeled compounds within the control limits specified? (Exception: native dichlorobiphenyls)?					
14. Were all toxic congeners uniquely resolved from non-toxic congeners?					
15. Was an ICV analyzed, calculated using the CS3 RRFs, and the %D within ± 35%)?				< 5 outliers, none more than ±50% D.	
16. If manual integrations were performed, are they clearly identified, initialed and dated?					
17. If criteria were not met, was an NCM generated, approved by supervisor, and copy included in folder?					
18. Does the ICAL folder contain **complete** data in the following order? Data review checklist, a **complete** run log, Avg. %RSD summary, Ratio summary, Calculation summary, PFK resolution/peak match documentation (HRMS only), and Total RIC, EICP's and manual integration - for window and all standards, in order from low to high standard.					

Analyst:	Date:	2nd Level Reviewer :	Date:
Comments:		**Comments:**	

*At reduced accelerating voltage

Figure 3- Example Data Review Checklist (Continued)

TestAmerica Knoxville Specialty Organics Group GC/MS Continuing Calibration Data Review Checklist
Method or SOP Number: KNOX-ID-0013 Revision 11

Start PFK:		End PFK:		CS3 Filename:		Inst:		ICAL Date:	

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd Level
1. Was the mass resolution documented at both the beginning and end of the 12 hour shift?					
2. Was the instrument resolution ≥8,000 throughout (≥ 10,000 for m/z 342.9792) and ≥10,000 in the center of each m/z range. PFK m/z 192.9888, *230.9856, and *280.9824? PFK m/z 268.9824, *292.9824, and *380.9760? PFK m/z 342.9792, *380.9760, and *430.9728? PFK m/z 404.9760, *442.9728, and *530.9665?					
3. Were the measured exact masses listed above within 5 ppm at reduced accelerating voltage?					
4. Was date/time of analysis verified between analysis header and logbook as correct?					
5. Were the MID switch points set to encompass the retention time windows of each congener group?					
6. Was the valley height less than 40% of the height of the shorter of the two peaks for the pair PCB 23 and PCB 34, and the pair PCB 182 and PCB 187					
7. Was the continuing calibration performed at the beginning of the 12 hour period after successful mass resolution and GC resolution performance check?					
8. Was the %D for all Toxic analytes within ± 30% for 1668A/B and ± 25% for 1668C? (PCB 81, 77, 123, 118, 114, 105, 126, 167, 156, 157, 169, 189) Was the %D for all LOC analytes within ± 30% for 1668A/B and ± 25% for 1668C? (PCB 1, 3, 4, 15, 19, 37, 54, 104, 155, 188, 202, 205, 206, 208, 209)					
9. Was the %D for all non-toxic/non-LOC analytes within ± 30% (for all versions of 1668)?					
10. Were the response factors calculated for each labeled standard and unlabeled native analyte using the SOP specified reference compound, quantitation ions, and formula.					
11. Were the absolute retention times of all labeled internal standards within ± 15 seconds of the retention times obtained during initial calibration?					
12. Are %D within ± 50% for all labeled internal standards (for 1668A/B) or -50/+45% (for 1668C) in the calibration?					
13. Are the %D within ± 50% for all labeled surrogate standards (for all versions of 1668) in the calibration?					
14. Are the %D within -40/+30% (for 1668A/B) or ± 25% (for 1668C) for all labeled cleanup standards in the calibration? **Note: for 1668C, PCB28L's lower limit can extend to -35%D.**					
15. Are all S/N ratios ≥10 for the GC signals in each EICP (extracted ion chromatographic profile) including internal standards?					
16. Are RRTs of all unlabled toxic/LOC analytes within their respective RRT limits?					
17. Are the ion abundance ratios for all labeled and unlabeled analytes within the specified control limits?					
18. If manual integrations were performed, are they clearly identified, initialed and dated?					
19. If criteria were not met, was a NCM generated, approved by supervisor, and copy included in folder?					
20. Does the CCAL folder contain **complete** data in the following order? Data review checklist, a **complete** run log, CCAL summary, Ratio summary, Calculation summary, PFK resolution/peak match documentation (HRMS only), and Total RIC, EICP's and manual integration - for window and both standards.					

Analyst:	Date:	2nd Level Reviewer :	Date:
Comments:		Comments:	

*At reduced accelerating voltag

Figure 3 - Example Data Review Checklist (Continued)

TestAmerica Knoxville SOG GC/MS Data Review/Narrative Checklist **Lot #**_____
SOP Number: KNOX-ID-0013 Revision 11 **Page 1 of 1** **Batch #**_____

Review Items **A. Initial Calibration**	N/A	Yes	No	Why is data reportable?	2nd
1. Was the correct ICAL used for quantitation? (Check 1-2 compounds for batch by manually calculating concentration using the ICAL avg. RF.)					
B. Continuing Calibration	N/A	Yes	No		2nd
1. Has a Continuing Calibration Checklist been completed for each analytical batch?					
C. Client Sample AND QC Sample Results	N/A	Yes	No		2nd
1. Were all special project requirements met?				See narrative	
2. Were the header information, prep factors, and dilution factors verified?					
3. Is logbook date/time of analysis correct?					
4. Sample analyses done within preparation and analytical holding time (HT)? *If no, list samples:* _____				☐ HT expired upon receipt. ☐ Client requested analysis after HT expired. ☐ Re-extraction done after HT expired.	
5. Are internal standards within QC limits? *If no, list samples and reason (e.g.,* **sur1***):* Sample Reason Sample Reason _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____				☐ **[sup]** Ion suppression due to matrix. ☐ **[low]** Low recovery. S/N >10 and EDL<ML. ☐ **[sam]** Not enough sample to re-extract. ☐ **[dil]** Dilution showed acceptable %R. ☐ **[mtx]** Obvious matrix interference. Further cleanup not possible.	
6. Were peaks ≥2.5 S/N, which did not meet the following criteria, properly calculated and reported as EMPCs? • All analytes within Method/SOP retention time criteria and both ions maximized within ±2 seconds. • The ion abundance ratios for all labeled and unlabeled analytes within the specified control limits.					
7. Are all results < the upper calibration level? *If no, list samples:* _____				☐ Sample extracted at lowest possible volume	
8. If manual integrations were performed, are they clearly identified, initialed and dated?					
9. Final report acceptable? (Results correct, DLs calculated correctly, units correct, IS %R correct, appropriate flags used, dilution factor correct, and extraction/ analysis dates correct.)					
10. Was a narrative prepared and all deviations noted?					
D. Preparation/Matrix QC	N/A	Yes	No	Why is data reportable?	2nd
1. LCS done per prep batch and all LCS/LCSD recoveries and RPDs within QC limits? *If no, list ID(s):* _____				☐ LCS/LCSD recoveries are high, no analytes detected above ML. ☐ Re-analysis not possible-insufficient sample ☐ See Comment/narrative	
2. Method blank done per prep batch and method blank or instrument blank analyzed with each sequence?					
3. Method blank internal standard recoveries within QC limits? *If no, list blank ID:* _____				☐ Internal standards are high and blank is free of contaminants. ☐ Internal standards are low, blank is free of contaminants, S/N>10 and EDL<EML.	
4. Are all analytes present in the method blank ≤ EML? *If no, list blank ID:* _____				☐ Sample results are > 20x higher than blank. ☐ No affected analytes > RL in the samples. ☐ Not enough sample for re-extraction.	
5. MS/MSD done per batch and are all recoveries and RPDs within laboratory generated QC limits? *If no, list MS/MSD ID:* _____				☐ LCS acceptable indicating sample matrix effects. ☐ LCS acceptable, high analyte concentration. ☐ LCS acceptable, lack of sample homogeneity.	
E. Other	N/A	Yes	No		2nd
1. Are all nonconformances documented appropriately and copy included with deliverable?					

Analyst:	**Date:**	**Analyst:**	**Date:**
Comments:		**Comments:**	