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EE/CA and RI/FS Support Sampling Plan

Sauget Area 1

Sauget and Cahokia, Illinois

Volume 2 - Appendix A

Soil, Groundwater, Surface Water, Sediment and Air FSP, QAPP and HASP

April 9, 1999

Submitted To:

U.S. Environmental Protection Agency
Chicago, Illinois

Submitted By:

Solutia Inc.

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,4,5-Trichlorophenol	8270(3550)	2	25-130	0-36	29	330
	CLP OLM01.9	62	NA	NA	800	800
	CLP OLM03.1	85	NA	NA	830	830
2,4,6-Trichlorophenol	8270(3550)	2	41-130	0-30	20	330
	CLP OLM01.9/OLM03.1	62/85	NA	NA	330	330
Trichlorophenols (2,4,5 + 2,4,6)	8270(3550)	2	NA	NA	NA	330
o,o,o-Triethylphosphorothioate	8270(3550)	2	10-150	0-50	51	330
1,3,5-Trinitrobenzene	8270(3550)	2	10-150	0-50	21	330
Surrogate – Nitrobenzene-d5	8270(3550)	2	20-120	NA	NA	NA
	CLP OLM01.9/OLM03.1	62/85	23-120	NA	NA	NA
Surrogate – 2-Fluorobiphenyl	8270(3550)	2	30-120	NA	NA	NA
	CLP OLM01.9/OLM03.1	62/85	30-115	NA	NA	NA
Surrogate – p-Terphenyl-d14	8270(3550)	2	30-131	NA	NA	NA
	CLP OLM01.9/OLM03.1	62/85	18-137	NA	NA	NA
Surrogate – Phenol-d5	8270(3550)	2	19-114	NA	NA	NA
	CLP OLM01.9/OLM03.1	62/85	23-113	NA	NA	NA
Surrogate – 2-Fluorophenol	8270(3550)	2	16-113	NA	NA	NA
	CLP OLM01.9/OLM03.1	62/85	25-121	NA	NA	NA
Surrogate – 2,4,6- Tribromophenol	8270(3550)	2	23-129	NA	NA	NA
	CLP OLM01.9/OLM03.1	62/85	19-122	NA	NA	NA
Surrogate – 2-Chlorophenol-d4	CLP OLM01.9/OLM03.1	62/85	20-130	NA	NA	NA
Surrogate -1,2-Dichlorobenzene-d4	CLP OLM01.9/OLM03.1	62/85	20-130	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Polychlorinated Dibenzo-p-dioxin and Dibenzofuran 2,3,7,8-substituted Congeners						
2,3,7,8-TCDD	8280	2	69-145	0-40	0.0069	0.50
2,3,7,8-TCDF	8280	2	59-142	0-40	0.062	0.50
1,2,3,7,8-PeCDF	8280	2	55-146	0-40	0.11	0.50
1,2,3,7,8-PeCDD	8280	2	41-203	0-40	0.12	0.50
2,3,4,7,8-PeCDF	8280	2	55-146	0-40	0.13	0.50
1,2,3,4,7,8-HxCDF	8280	2	50-154	0-46	0.10	0.50
1,2,3,6,7,8-HxCDF	8280	2	50-154	0-46	0.13	0.50
1,2,3,4,7,8-HxCDD	8280	2	45-174	0-53	0.12	0.50
1,2,3,6,7,8-HxCDD	8280	2	45-174	0-53	0.12	0.50
1,2,3,7,8,9-HxCDD	8280	2	45-174	0-53	0.12	0.50
2,3,4,6,7,8-HxCDF	8280	2	50-154	0-46	0.057	0.50
1,2,3,7,8,9-HxCDF	8280	2	50-154	0-46	0.057	0.50
1,2,3,4,7,8,9-HpCDF	8280	2	20-170	0-50	0.062	1.0
1,2,3,4,6,7,8-HpCDF	8280	2	20-170	0-50	0.065	1.0
1,2,3,4,6,7,8-HpCDD	8280	2	20-170	0-50	0.16	1.0
1,2,3,4,5,6,7,8-octa-CDD (MS)	8280	2	20-170	0-50	0.14	1.0
1,2,3,4,5,6,7,8-octa-CDF (MS)	8280	2	20-170	0-50	0.13	1.0
Polychlorinated Dibenzo-p-dioxin and Dibenzofuran classes						
tetra-CDD (MS)	8280	2	69-145	0-40	0.12	0.50
tetra-CDF (MS)	8280	2	59-142	0-40	0.040	0.50
penta-CDD (MS)	8280	2	41-203	0-40	0.089	0.50
Penta-CDF (MS)	8280	2	55-146	0-40	0.068	0.50
hexa-CDD (MS)	8280	2	45-174	0-53	0.13	0.50
hexa-CDF (MS)	8280	2	50-154	0-46	0.051	0.50
hepta-CDD (MS)	8280	2	20-170	0-50	0.13	1.0
hepta-CDF (MS)	8280	2	20-170	0-50	0.063	1.0
Internal Standards						
2,3,7,8-tetra-CDD-13C12	8280	2	25-150	NA	NA	NA
2,3,7,8-tetra-CDF-13C12	8280	2	25-150	NA	NA	NA
1,2,3,6,7,8-hexa-CDD-13C12	8280	2	25-150	NA	NA	NA
1,2,3,4,6,7,8-hepta-CDF-13C12	8280	2	25-150	NA	NA	NA
octa-CDD-13C12	8280	2	25-150	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Chlordimeform (Galecron)	625(3550)	1/2	10-150	0-50	580	670
5-Chlordimeform	625(3550)	1/2	10-150	0-50	300	330
Dithiocarbamates (as Ziram)[5]	630(3540)	63/2	50-126	0-52	190	1000
Benomyl (as Carbendazim)	631	55/2	49-139	0-50	7.8	30
Aminocarb	632(3550)	13/2	50-150	0-50	2.2	50
Barban	632(3550)	13/2	85-128	0-25	8.3	20
Bromacil	632(3550)	13/2	66-120	0-25	14	40
Carbaryl (MS)	632(3550)	13/2	50-150	0-50	14	50
Carbofuran	632(3550)	13/2	23-109	0-31	23	50
Chlorpropham	632(3550)	13/2	50-150	0-50	11	20
Diuron (MS)	632(3550)	13/2	50-150	0-50	3.3	5.0
Fenuron	632(3550)	13/2	50-150	0-50	4.1	10
Fluometuron	632(3550)	13/2	50-150	0-50	5.0	10
Linuron	632(3550)	13/2	64-130	0-28	3.8	5.0
Methiocarb	632(3550)	13/2	50-150	0-50	13	50
Methomyl	632(3550)	13/2	50-150	0-50	28	200
Monuron	632(3550)	13/2	65-109	0-25	2.7	5.0
Neburon	632(3550)	13/2	50-150	0-50	2.9	5.0
Oxamyl	632(3550)	13/2	50-150	0-50	12	50
Propham	632(3550)	13/2	50-150	0-50	10	50
Propoxur	632(3550)	13/2	63-116	0-33	69	200
Siduron	632(3550)	13/2	50-150	0-50	15	20
Swep	632(3550)	13/2	50-150	0-50	4.9	20
Oryzalin	638(3550)	21/2	60-140	0-40	4.5	25

[5]The compounds determined as ziram by Method 630 include amobam, farbam, mancozeb, maneb, metham, nabam, polyram, and zineb.

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Acenaphthene (MS)	8310(3550)	2	11-144	0-35	8.3	50
Acenaphthylene	8310(3550)	2	10-139	0-40	8.7	20
Anthracene	8310(3550)	2	10-126	0-40	0.34	4.0
Benzo(a)anthracene	8310(3550)	2	12-135	0-40	1.2	4.0
Benzo(b)fluoranthene	8310(3550)	2	10-150	0-40	0.27	4.0
Benzo(k)fluoranthene	8310(3550)	2	10-159	0-40	0.54	4.0
Benzo(g,h,i)perylene	8310(3550)	2	10-120	0-40	1.4	10
Benzo(a)pyrene	8310(3550)	2	10-128	0-40	1.0	4.0
Chrysene (MS)	8310(3550)	2	10-199	0-40	1.0	4.0
Dibenzo(a,h)anthracene	8310(3550)	2	10-110	0-40	3.7	10
Fluoranthene	8310(3550)	2	56-136	0-28	1.4	10
Fluorene (MS)	8310(3550)	2	10-142	0-40	0.82	10
Indeno(1,2,3-cd)pyrene	8310(3550)	2	10-116	0-40	1.1	10
1-Methylnaphthalene	8310(3550)	2	10-125	0-40	12	20
2-Methylnaphthalene	8310(3550)	2	10-125	0-40	5.1	20
Naphthalene (MS)	8310(3550)	2	31-159	0-34	6.0	20
Phenanthrene	8310(3550)	2	10-155	0-40	0.77	4.0
Pyrene (MS)	8310(3550)	2	49-156	0-28	2.1	10
Surrogate - 4-Terphenyl-d14	8310(3550)	2	28-151	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL A (ug/kg)
Acetaldehyde	8315	2	30-156	0-36	19	250
Formaldehyde	8315	2	30-170	0-47	23	100
Acrylamide	8316	2	27-120	0-50	220	1000
Aldicarb (Temik) (MS)	8318	2	44-114	0-50	0.79	10
Aldicarb sulfone	8318	2	58-118	0-50	1.0	10
Aldicarb sulfoxide	8318	2	33-143	0-50	1.0	10
Carbofuran (Furadan) (MS)	8318	2	53-123	0-50	1.7	10
Carbaryl (Sevin)	8318	2	56-126	0-50	1.6	10
Dioxacarb	8318	2	55-125	0-50	1.9	10
3-Hydroxycarbofuran	8318	2	60-120	0-50	1.6	10
Methiocarb (Mesurol)	8318	2	52-122	0-50	2.0	10
Methomyl (Lannate)	8318	2	54-114	0-50	1.7	10
Oxamyl (MS)	8318	2	45-161	0-50	1.8	10
Promecarb	8318	2	44-120	0-50	1.7	10
Propoxur (Baygon)	8318	2	46-116	0-50	1.5	10

TABLE 5.2 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2-Amino-4,6-dinitrotoluene	8330	2	50-150	0-30	130	250
4-Amino-2,6-dinitrotoluene	8330	2	58-126	0-30	150	500
1,3-Dinitrobenzene (MS)	8330	2	50-132	0-30	39	250
2,4-Dinitrotoluene (MS)	8330	2	54-120	0-30	39	250
2,6-Dinitrotoluene	8330	2	59-122	0-30	87	500
Diphenylamine	8330	2	65-140	0-30	16	100
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	8330	2	54-166	0-30	99	500
Methyl-2,4,6-trinitro-phenylnitramine (Tetryl)	8330	2	41-165	0-30	220	500
Nitrobenzene	8330	2	52-152	0-30	130	250
Nitroglycerin	8332	2	48-152	0-50	38	1000
n-Nitrosodiphenylamine	8330	2	55-121	0-30	32	100
2-Nitrotoluene (MS)	8330	2	52-138	0-40	77	250
3-Nitrotoluene	8330	2	69-137	0-30	140	250
4-Nitrotoluene	8330	2	54-166	0-30	130	250
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	8330	2	29-156	0-600	130	500
Pentaerythritol tetranitrate (PETN)	8330	2	50-150	0-30	34	1000
1,3,5-Trinitrobenzene	8330	2	47-123	0-30	50	250
2,4,6-Trinitrotoluene	8330	2	74-119	0-30	74	250
Surrogate - 3,4-Dinitrotoluene	8330	2	22-128	A	NA	NA

TABLE 5.2 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Glyphosate	547/SL SOP	51	50-150	0-50	570	2500
Acrylic Acid	SL-SOP	68	25-150	0-50	1100	5000
Cyanuric acid	SL-SOP	102	50-150	0-50	150	2500
Ethylenethiourea	SL-SOP	104	40-120	0-40	7.6	50
Maleic Acid/Maleic Anhydride	SL-SOP	103	69-106	0-21	20	80
Nitrocellulose	SL-SOP	108	20-150	0-60	14000	50000
Phthalic acid/Phthalic anhydride	SL-SOP	103	30-142	0-40	5.6	100
Thiodiglycol	SL-SOP	112	40-128	0-50	400	1500

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Arsenic	1311/6010(3010)	2	75-125	0-20	0.0053	0.20
Barium	1311/6010(3010)	2	75-125	0-20	0.0012	1.0
Cadmium	1311/6010(3010)	2	75-125	0-20	0.00071	0. 10
Chromium	1311/6010(3010)	2	75-125	0-20	0.0017	0.20
Lead	1311/6010(3010)	2	75-125	0-20	0.0015	0.20
Mercury	1311/7470	2	80-120	0-20	0.0072	0.020
Selenium	1311/6010(3010)	2	75-125	0-20	0.0042	0.50
Silver	1311/6010(3010)	2	75-125	0-20	0.0019	0.10

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
ndrin	1311/8081 (3510/3520)	2	41-158	0-25	0.00023	0.0050
Lindane	1311/8081 (3510/3520)	2	40-139	0-36	0.000052	0.0025
Methoxychlor	1311/8081 (3510/3520)	2	60-155	0-43	0.0016	0.025
Chlordane	1311/8081 (3510/3520)	2	54-140	0-30	0.0061	0.025
Toxaphene	1311/8081 (3510/3520)	2	12-130	0-30	0.062	0.25
Heptachlor	1311/8081 (3510/3520)	2	37-148	0-26	0.00011	0.0025
Heptachlor Epoxide	1311/8081 (3510/3520)	2	43-141	0-31	0.000088	0.0025
Surrogate - Tetrachloro-m-xylene (TCMX)	1311/8081 (3510/3520)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	1311/8081 (3510/3520)	2	30-150	NA	NA	NA
Surrogate - Dibutyl chlorendate (DBC)	1311/8081 (3510/3520)	2	30-150	NA	NA	NA

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
2,4-D	1311/8151	2	11-154	0-78	0.0095	0.025
2,4,5-TP (Silvex)	1311/8151	2	10-100	0-66	0.00022	0.025
Surrogate - DCAA	1311/8151	2	27-133	NA	NA	NA

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Benzene	1311/8260 (5030)	2	62-135	0-16	0.0011	0.020
Carbon tetrachloride	1311/8260 (5030)	2	57-128	0-13	0.0021	0.020
Chlorobenzene	1311/8260 (5030)	2	72-127	0-22	0.0032	0.020
1,2-Dichloroethane	1311/8260 (5030)	2	65-131	0-23	0.0023	0.020
Chloroform	1311/8260 (5030)	2	62-130	0-20	0.0036	0.020
1,1-Dichloroethylene	1311/8260 (5030)	2	46-147	0-30	0.0018	0.020
Methylethyl ketone	1311/8260 (5030)	2	42-167	0-31	0.044	0.10
Trichloroethylene	1311/8260 (5030)	2	56-143	0-35	0.0011	0.020
Tetrachloroethylene	1311/8260 (5030)	2	60-148	0-24	0.0064	0.020
Vinyl chloride	1311/8260 (5030)	2	43-142	0-21	0.0048	0.040
Surrogate - Toluene-d8	1311/8260 (5030)	2	72-122	NA	NA	NA
Surrogate - p-Bromofluorobenzene	1311/8260 (5030)	2	74-126	NA	NA	NA
Surrogate - Dibromofluoromethane	1311/8260 (5030)	2	70-130	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	1311/8260 (5030)	2	70-130	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	1311/8260 (5030)	2	70-130	NA	NA	NA

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Nitrobenzene	1311/8270 (3510/3520)	2	50-111	0-21	0.0016	0.050
Hexachlorobenzene	1311/8270 (3510/3520)	2	49-121	0-31	0.0095	0.050
1,4-Dichlorobenzene	1311/8270 (3510/3520)	2	27-103	0-31	0.0014	0.050
2,4-Dinitrotoluene	1311/8270 (3510/3520)	2	37-129	0-32	0.0020	0.050
Hexachlorobutadiene	1311/8270 (3510/3520)	2	27-97	0-30	0.0018	0.050
Hexachloroethane	1311/8270 (3510/3520)	2	26-86	0-35	0.0016	0.050
Pyridine	1311/8270 (3510/3520)	2	10-134	0-50	0.0046	0.25
2,4,5-Trichlorophenol	1311/8270 (3510/3520)	2	38-127	0-28	0.0037	0.050
2,4,6-Trichlorophenol	1311/8270 (3510/3520)	2	36-126	0-22	0.0018	0.050
Cresols	1311/8270 (3510/3520)	2	24-136	0-27	0.0036	0.050
Pentachlorophenol	1311/8270 (3510/3520)	2	19-148	0-33	0.020	0.25
Surrogate - Nitrobenzene-d5	1311/8270 (3510/3520)	2	34-130	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	1311/8270 (3510/3520)	2	36-124	NA	NA	NA
Surrogate - p-Terphenyl-d14	1311/8270 (3510/3520)	2	14-148	NA	NA	NA
Surrogate - Phenol-d5	1311/8270 (3510/3520)	2	25-128	NA	NA	NA
Surrogate - 2-Fluorophenol	1311/8270 (3510/3520)	2	29-121	NA	NA	NA
Surrogate - 2,4,6-Tribromophenol	1311/8270 (3510/3520)	2	29-143	NA	NA	NA

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Dichlorodifluoromethane	TO1/TO2	75	60-140	0-40	0.05	0.20
Chloromethane	TO1/TO2	75	60-140	0-40	0.04	0.20
Vinyl chloride	TO1/TO2	75	60-140	0-40	0.08	0.20
Bromomethane	TO1/TO2	75	60-140	0-40	0.04	0.20
Chloroethane	TO1/TO2	75	60-140	0-40	0.07	0.20
Trichlorofluoromethane	TO1/TO2	75	60-140	0-40	0.04	0.20
1,1-Dichloroethene	TO1/TO2	75	60-140	0-40	0.02	0.10
Methylene chloride (Dichloromethane)	TO1/TO2	75	60-140	0-40	0.01	0.10
Trans-1,2-Dichloroethene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,1-Dichloroethane	TO1/TO2	75	60-140	0-40	0.01	0.10
2,2-Dichloropropane	TO1/TO2	75	60-140	0-40	0.02	0.10
Cis-1,2-Dichloroethene	TO1/TO2	75	60-140	0-40	0.01	0.10
Chloroform	TO1/TO2	75	60-140	0-40	0.01	0.10
Bromochloromethane	TO1/TO2	75	60-140	0-40	0.01	0.10
1,1,1-Trichloroethane	TO1/TO2	75	60-140	0-40	0.01	0.10
1,1-Dichloropropylene	TO1/TO2	75	60-140	0-40	0.02	0.10
Carbon tetrachloride	TO1/TO2	75	60-140	0-40	0.01	0.10
1,2-Dichloroethane	TO1/TO2	75	60-140	0-40	0.01	0.10
Benzene	TO1/TO2	75	60-140	0-40	0.01	0.10
Trichloroethylene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,2-Dichloropropane	TO1/TO2	75	60-140	0-40	0.01	0.10
Bromodichloromethane	TO1/TO2	75	60-140	0-40	0.03	0.10
Dibromomethane	TO1/TO2	75	60-140	0-40	0.02	0.10
Trans-1,3-Dichloropropene	TO1/TO2	75	60-140	0-40	0.01	0.10
Toluene	TO1/TO2	75	60-140	0-40	0.01	0.10
Cis-1,3-Dichloropropene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,1,2-Trichloroethane	TO1/TO2	75	60-140	0-40	0.01	0.10
1,3-Dichloropropane	TO1/TO2	75	60-140	0-40	0.02	0.10
Tetrachloroethene	TO1/TO2	75	60-140	0-40	0.01	0.10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Dibromochloromethane	TO1/TO2	75	60-140	0-40	0.02	0.10
1,2-Dibromomethane	TO1/TO2	75	60-140	0-40	0.02	0.10
Chlorobenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,1,1,2-Tetrachloroethane	TO1/TO2	75	60-140	0-40	0.02	0.10
Ethylbenzene	TO1/TO2	75	60-140	0-40	0.06	0.10
M&p-Xylene	TO1/TO2	75	60-140	0-40	0.01	0.10
o-Xylene	TO1/TO2	75	60-140	0-40	0.01	0.10
Styrene	TO1/TO2	75	60-140	0-40	0.01	0.10
Isopropylbenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
Bromoform	TO1/TO2	75	60-140	0-40	0.01	0.10
1,1,2,2-Tetrachloroethane	TO1/TO2	75	60-140	0-40	0.01	0.10
1,2,3-Trichloropropane	TO1/TO2	75	60-140	0-40	0.01	0.10
n-Propylbenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
Bromobenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,3,5-Trimethylbenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
2-Chlorotoluene	TO1/TO2	75	60-140	0-40	0.01	0.10
4-Chlorotoluene	TO1/TO2	75	60-140	0-40	0.01	0.10
t-Butylbenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,2,4-Trimethylbenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
s-Butylbenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
p-Isopropyltoluene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,3-Dichlorobenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,4-Dichlorobenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
n-Butylbenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,2-Dichlorobenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
1,2-Dibromo-3-chloropropane	TO1/TO2	75	60-140	0-40	0.02	0.10
1,2,4-Trichlorobenzene	TO1/TO2	75	60-140	0-40	0.01	0.10
Hexachlorobutadiene	TO1/TO2	75	60-140	0-40	0.02	0.10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Naphthalene	TO1/TO2	75	60-140	0-40	0.02	0.10
1,2,3-Trichlorobenzene	TO1/TO2	75	60-140	0-40	0.02	0.10
Acetone	TO1/TO2	75	60-140	0-40	0.21	1.0
2-Butanone (MEK)	TO1/TO2	75	60-140	0-40	0.09	1.0
Vinyl Acetate	TO1/TO2	75	60-140	0-40	0.01	0.10
4-Methyl-2-pentanone (MIBK)	TO1/TO2	75	60-140	0-40	0.16	1.0
2-Hexanone	TO1/TO2	75	60-140	0-40	0.09	1.0
Carbon disulfide	TO1/TO2	75	60-140	0-40	0.02	0.10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Foramaldehyde	TO5	75	43-142	0-30	3.5	50
Formaldehyde	TO11	75	43-142	0-30	3.5	50

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Aldrin	TO4/TO10	75	10-144	0-38	0.0041	0.050
alpha-BHC	TO4/TO10	75	22-101	0-40	0.0022	0.050
beta-BHC	TO4/TO10	75	12-120	0-40	0.0053	0.050
Gamma-BHC (Lindane)	TO4/TO10	75	12-138	0-37	0.0019	0.050
delta-BHC	TO4/TO10	75	10-142	0-47	0.0019	0.050
Technical Chlordane	TO4/TO10	75	45-119	0-40	0.061	0.50
alpha Chlordane	TO4/TO10	75	45-140	0-40	0.0028	0.050
Gamma Chlordane	TO4/TO10	75	11-141	0-40	0.0026	0.050
4,4'-DDD	TO4/TO10	75	28-134	0-50	0.0060	0.10
4,4'-DDE	TO4/TO10	75	34-121	0-23	0.0069	0.10
4,4'-DDT	TO4/TO10	75	29-134	0-26	0.0074	0.10
Dieldrin	TO4/TO10	75	28-137	0-30	0.0023	0.10
Endosulfan I	TO4/TO10	75	10-141	0-40	0.0027	0.050
Endosulfan II	TO4/TO10	75	10-141	0-65	0.0037	0.10
Endosulfan sulfate	TO4/TO10	75	26-144	0-50	0.0053	0.10
rin	TO4/TO10	75	33-149	0-32	0.0085	0.10
drin aldehyde	TO4/TO10	75	10-130	0-86	0.0079	0.10
ndrin ketone	TO4/TO10	75	29-112	0-31	0.011	0.10
Heptachlor	TO4/TO10	75	17-138	0-38	0.0025	0.050
Heptachlor epoxide	TO4/TO10	75	15-142	0-40	0.0085	0.050
Methoxychlor	TO4/TO10	75	24-152	0-40	0.032	0.50
Toxaphene	TO4/TO10	75	41-126	0-50	0.55	5.0
PCB-1016	TO4/TO10	75	34-137	0-44	0.15	1.0
PCB 1221	TO4/TO10	75	15-178	0-30	0.27	1.0
PCB 1232	TO4/TO10	75	10-215	0-30	0.21	1.0
PCB-1242	TO4/TO10	75	39-150	0-30	0.25	1.0
PCB-1248	TO4/TO10	75	38-158	0-30	0.18	1.0
PCB-1254	TO4/TO10	75	66-122	0-30	0.11	1.0
PCB-1260	TO4/TO10	75	58-150	0-30	0.12	1.0
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	TO4/TO10	75	10-114	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	TO4/TO10	75	27-128	NA	NA	NA

TABLE 5.4. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Acenaphthene	TO13	75	67-112	0-25	4.4	10
Acenaphthylene	TO13	75	60-102	0-25	4.1	10
Anthracene	TO13	75	70-130	0-25	3.8	10
Benzo(a)anthracene	TO13	75	73-101	0-25	2.7	10
Benzo(b)fluoranthene	TO13	75	70-130	0-25	3.0	10
Benzo(k)fluoranthene	TO13	75	70-130	0-25	4.9	10
Benzo(a)pyrene	TO13	75	74-104	0-25	2.9	10
Benzo(g,h,i)perylene	TO13	75	48-100	0-25	2.2	10
Benzyl alcohol	TO13	75	58-100	0-25	2.4	10
bis(2-Chloroethoxy)methane	TO13	75	66-100	0-25	2.5	10
bis(2-Chloroethyl)ether	TO13	75	50-100	0-25	2.2	10
bis(2-Chloroisopropyl)ether	TO13	75	62-100	0-25	3.3	10
bis(2-Ethylhexyl)phthalate	TO13	75	50-150	0-25	4.9	10
4-Bromophenyl phenyl ether	TO13	75	70-130	0-25	3.9	10
Butylbenzyl phthalate	TO13	75	50-150	0-25	2.8	10
4-Chloroaniline	TO13	75	62-100	0-25	2.6	20
4-Chloro-3-methyl phenol	TO13	75	70-130	0-25	2.6	10
2-chloronapthalene	TO13	75	69-100	0-25	2.8	10
2-Chlorophenol	TO13	75	53-100	0-25	2.7	10
4-Chlorophenyl phenyl ether	TO13	75	50-150	0-25	4.2	10
Chrysene	TO13	75	70-130	0-25	2.4	10
Dibenz(a,h)anthracene	TO13	75	55-100	0-25	2.4	10
Dibenzofuran	TO13	75	74-122	0-25	4.7	10
1,2-Dichlorobenzene	TO13	75	46-100	0-25	3.4	10
1,3-Dichlorobenzene	TO13	75	41-100	0-25	3.1	10
1,4-Dichlorobenzene	TO13	75	44-100	0-25	3.3	10
3,3'-Dichlorobenzidine	TO13	75	77-136	0-25	5.6	10
2,4-Dichlorophenol	TO13	75	69-100	0-25	2.7	10
Diethylphthalate	TO13	75	70-130	0-25	3.4	10

TABLE 5.4. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
2,4-Dimethylphenol	TO13	75	48-100	0-25	2.5	10
Dimethylphthalate	TO13	75	70-130	0-25	2.9	10
Di-n-butylphthalate	TO13	75	50-150	0-25	2.0	10
2,4-Dinitrotoluene	TO13	75	66-110	0-25	4.2	10
2,6-Dinitrotoluene	TO13	75	59-100	0-25	3.2	10
Di-n-octyl phthalate	TO13	75	50-150	0-25	5.9	10
Fluoranthene	TO13	75	70-130	0-25	3.8	10
Fluorene	TO13	75	70-130	0-25	5.1	10
Hexachlorobenzene	TO13	75	74-123	0-25	4.7	10
Hexachlorobutadiene	TO13	75	58-104	0-25	4.4	10
01Hexachloroethane	TO13	75	44-100	0-25	2.5	10
Indeno(1,2,3-cd)pyrene	TO13	75	59-100	0-25	2.6	10
Isophorone	TO13	75	52-100	0-25	3.9	10
2-Methyl naphthalene	TO13	75	24-107	0-25	8.0	10
2-Methylphenol	TO13	75	59-100	0-25	3.0	10
4-Methylphenol	TO13	75	59-109	0-25	4.8	10
Naphthalene	TO13	75	30-100	0-25	6.5	10
2-Nitroaniline	TO13	75	62-100	0-25	2.5	50
3-Nitroaniline	TO13	75	75-100	0-25	2.4	50
4-Nitroaniline	TO13	75	76-100	0-25	2.0	50
Nitrobenzene	TO13	75	20-100	0-25	5.0	10
2-Nitrophenol	TO13	75	35-100	0-25	2.4	10
N-nitrosodiphenylamine	TO13	75	70-130	0-25	2.8	10
N-nitrosodi-n-propylamine	TO13	75	63-100	0-25	3.0	10
Phenanthrene	TO13	75	70-130	0-25	3.6	10
Phenol	TO13	75	74-135	0-25	5.8	10
Pyrene	TO13	75	70-130	0-25	3.7	10
1,2,4-Trichlorobenzene	TO13	75	57-100	0-25	3.8	10
2,4,5-Trichlorophenol	TO13	75	77-121	0-25	4.2	10
2,4,6-Trichlorophenol	TO13	75	65-105	0-25	3.8	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m³)	RLA (mg/m³)
Dichlorodifluoromethane	TO14/TO15	75	70-130%	0-30	0.090	0.20
Chloromethane	TO14/TO15	75	70-130%	0-30	0.13	0.20
Vinyl chloride	TO14/TO15	75	70-130%	0-30	0.10	0.20
Bromomethane	TO14/TO15	75	70-130%	0-30	0.050	0.20
Chloroethane	TO14/TO15	75	70-130%	0-30	0.070	0.20
Trichlorofluoromethane	TO14/TO15	75	70-130%	0-30	0.060	0.20
1,1-Dichloroethene	TO14/TO15	75	70-130%	0-30	0.040	0.10
Methylene chloride (Dichloromethane)	TO14/TO15	75	70-130%	0-30	0.060	0.10
Trans-1,2-Dichloroethene	TO14/TO15	75	70-130%	0-30	0.040	0.10
1,1-Dichloroethane	TO14/TO15	75	70-130%	0-30	0.050	0.10
2,2-Dichloropropane	TO14/TO15	75	70-130%	0-30	0.030	0.10
Cis-1,2-Dichloroethene	TO14/TO15	75	70-130%	0-30	0.040	0.10
Chloroform	TO14/TO15	75	70-130%	0-30	0.050	0.10
Bromochloromethane	TO14/TO15	75	70-130%	0-30	0.060	0.10
1,1,1-Trichloroethane	TO14/TO15	75	70-130%	0-30	0.040	0.10
1,1-Dichloropropylene	TO14/TO15	75	70-130%	0-30	0.040	0.10
Carbon tetrachloride	TO14/TO15	75	70-130%	0-30	0.040	0.10
1,2-Dichloroethane	TO14/TO15	75	70-130%	0-30	0.040	0.10
Benzene	TO14/TO15	75	70-130%	0-30	0.040	0.10
Trichloroethylene	TO14/TO15	75	70-130%	0-30	0.040	0.10
1,2-Dichloropropane	TO14/TO15	75	70-130%	0-30	0.040	0.10
Bromodichloromethane	TO14/TO15	75	70-130%	0-30	0.040	0.10
Dibromomethane	TO14/TO15	75	70-130%	0-30	0.040	0.10
Trans-1,3-Dichloropropene	TO14/TO15	75	70-130%	0-30	0.050	0.10
Toluene	TO14/TO15	75	70-130%	0-30	0.040	0.10
Cis-1,3-Dichloropropene	TO14/TO15	75	70-130%	0-30	0.040	0.10
1,1,2-Trichloroethane	TO14/TO15	75	70-130%	0-30	0.050	0.10
1,3-Dichloropropane	TO14/TO15	75	70-130%	0-30	0.050	0.10
Tetrachloroethene	TO14/TO15	75	70-130%	0-30	0.050	0.10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m³)	RLA (mg/m³)
Dibromochloromethane	TO14/TO15	75	70-130%	0-30	0.050	0.10
1,2-Dibromoethane(EDB)	TO14/TO15	75	70-130%	0-30	0.050	0.10
Chlorobenzene	TO14/TO15	75	70-130%	0-30	0.040	0.10
1,1,1,2-Tetrachloroethane	TO14/TO15	75	70-130%	0-30	0.040	0.10
Ethylbenzene	TO14/TO15	75	70-130%	0-30	0.040	0.10
m&p-Xylene	TO14/TO15	75	70-130%	0-30	0.040	0.10
o-Xylene	TO14/TO15	75	70-130%	0-30	0.050	0.10
Styrene	TO14/TO15	75	70-130%	0-30	0.050	0.10
Isopropylbenzene	TO14/TO15	75	70-130%	0-30	0.040	0.10
Bromoform	TO14/TO15	75	70-130%	0-30	0.050	0.10
1,1,2,2-Tetrachloroethane	TO14/TO15	75	70-130%	0-30	0.050	0.10
1,2,3-Trichloropropane	TO14/TO15	75	70-130%	0-30	0.040	0.10
n-Propylbenzene	TO14/TO15	75	70-130%	0-30	0.040	0.10
Bromobenzene	TO14/TO15	75	70-130%	0-30	0.050	0.10
1,3,5-Trimethylbenzene	TO14/TO15	75	70-130%	0-30	0.040	0.10
2-Chlorotoluene	TO14/TO15	75	70-130%	0-30	0.040	0.10
4-Chlorotoluene	TO14/TO15	75	70-130%	0-30	0.050	0.10
t-Butylbenzene	TO14/TO15	75	70-130%	0-30	0.040	0.10
1,2,4-Trimethylbenzene	TO14/TO15	75	70-130%	0-30	0.050	0.10
s-Butylbenzene	TO14/TO15	75	70-130%	0-30	0.040	0.10
p-Isopropyltoluene	TO14/TO15	75	70-130%	0-30	0.040	0.10
1,3-Dichlorobenzene	TO14/TO15	75	70-130%	0-30	0.060	0.10
1,4-Dichlorobenzene	TO14/TO15	75	70-130%	0-30	0.070	0.10
n-Butylbenzene	TO14/TO15	75	70-130%	0-30	0.060	0.10
1,2-Dichlorobenzene	TO14/TO15	75	70-130%	0-30	0.060	0.10
1,2-Dibromo-3-chloropropane	TO14/TO15	75	70-130%	0-30	0.12	0.50
1,2,4-Trichlorobenzene	TO14/TO15	75	70-130%	0-30	0.21	0.50
Hexachlorobutadiene	TO14/TO15	75	70-130%	0-30	0.060	0.50

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m^3)	RLA (mg/m^3)
Naphthalene	TO14/TO15	75	70-130	0-30	0.50	0.50
1,2,3-trichlorobenzene	TO14/TO15	75	70-130	0-30	0.41	0.50
Acetone	TO14/TO15	75	70-130	0-30	0.61	1.0
2-Butanone (MEK)	TO14/TO15	75	70-130	0-30	0.18	1.0
Vinyl acetate	TO14/TO15	75	70-130	0-30	0.050	0.50
4-Methyl-2-pentanone (MIBK)	TO14/TO15	75	70-130	0-30	0.090	1.0
2-Hexanone	TO14/TO15	75	70-130	0-30	0.18	1.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m^3)	RLA (mg/m^3)
Dichlorodifluoromethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.77	2.0
Chloromethane	EPA 18 (Tedlar bag)	76	60-140	0-30	1.0	2.0
Vinyl chloride	EPA 18 (Tedlar bag)	76	60-140	0-30	0.51	2.0
Bromomethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.67	2.0
Chloroethane	EPA 18 (Tedlar bag)	76	60-140	0-30	1.0	2.0
Trichlorofluoromethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.51	1.0
1,1-Dichloroethene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.44	1.0
Methylene chloride (Dichloromethane)	EPA 18 (Tedlar bag)	76	60-140	0-30	0.26	1.0
Trans-1,2-Dichloroethene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.25	1.0
1,1-Dichloroethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.16	1.0
2,2-Dichloropropane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.25	1.0
Cis-1,2-Dichloroethene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.24	1.0
Chloroform	EPA 18 (Tedlar bag)	76	60-140	0-30	0.14	1.0
Bromochloromethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.25	1.0
1,1,1-Trichloroethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.26	1.0
1,1-Dichloropropylene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.20	1.0
Carbon tetrachloride	EPA 18 (Tedlar bag)	76	60-140	0-30	0.27	1.0
1,2-Dichloroethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.28	1.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m³)	RLA (mg/m³)
Benzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.13	1.0
Trichloroethylene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.15	1.0
1,2-Dichloropropane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.11	1.0
Bromodichloromethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.13	1.0
Dibromomethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.14	1.0
Trans-1,3-Dichloropropene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.15	1.0
Toluene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.21	1.0
Cis-1,3-Dichloropropene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.10	1.0
1,1,2-Trichloroethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.15	1.0
1,3-Dichloropropane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.12	1.0
Tetrachloroethene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.28	1.0
Dibromochloromethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.21	1.0
1,2-Dibromoethane(EDB)	EPA 18 (Tedlar bag)	76	60-140	0-30	0.13	1.0
Chlorobenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.12	1.0
Ethylbenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.18	1.0
m&p-Xylene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.44	1.0
o-Xylene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.27	1.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m^3)	RLA (mg/m^3)
Styrene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.20	1.0
Isopropylbenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.20	1.0
Bromoform	EPA 18 (Tedlar bag)	76	60-140	0-30	0.28	1.0
1,1,2,2-Tetrachloroethane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.24	1.0
1,2,3-Trichloropropane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.84	2.0
n-Propylbenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.19	1.0
Bromobenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.17	1.0
1,3,5-Trimethylbenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.22	1.0
2-Chlorotoluene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.36	1.0
4-Chlorotoluene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.22	1.0
t-Butylbenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.20	1.0
1,2,4-Trimethylbenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.27	1.0
s-Butylbenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.23	1.0
p-Isopropyltoluene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.23	1.0
1,3-Dichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.17	1.0
1,4-Dichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.17	1.0
n-Butylbenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.24	1.0
1,2-Dichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.16	1.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m³)	RLA (mg/m³)
1,2-Dibromo-3-chloropropane	EPA 18 (Tedlar bag)	76	60-140	0-30	0.87	2.0
1,2,4-Trichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.21	1.0
Hexachlorobutadiene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.25	1.0
Naphthalene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.32	1.0
1,2,3-Trichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	0-30	0.28	1.0
Acetone	EPA 18 (Tedlar bag)	76	60-140	0-30	0.52	2.0
2-Butanone (MEK)	EPA 18 (Tedlar bag)	76	60-140	0-30	1.5	2.0
Vinyl Acetate	EPA 18 (Tedlar bag)	76	60-140	0-30	0.59	2.0
4-Methyl-2-pentanone (MIBK)	EPA 18 (Tedlar bag)	76	60-140	0-30	0.64	2.0
2-Hexanone	EPA 18 (Tedlar bag)	76	60-140	0-30	0.94	2.0
Carbon disulfide	EPA 18 (Tedlar bag)	76	60-140	0-30	0.20	1.0
Methyl t-butyl ether (MTBE)	EPA 18 (Tedlar bag)	76	60-140	0-30	0.19	1.0
Total hydrocarbons	EPA 18 (Tedlar bag)	76	60-140	0-30	1.0	1.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	M ETHOD (prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (mg/m3)	RL (mg/m3)
Nitrogen(TCD)	EPA 18 (Tedlar bag)	76	75-125	0-25	10000	10000
Oxygen(TCD)	EPA 18 (Tedlar bag)	76	75-125	0-25	10000	10000
Carbon dioxide(TCD)	EPA 18 (Tedlar bag)	76	75-125	0-25	10000	18000
Carbon monoxide(TCD).	EPA 18 (Tedlar bag)	76	75-125	0-25	100	570
Methane(TCD)	EPA 18 (Tedlar bag)	76	75-125	0-25	100	330
Methane (FID)	EPA 18 (Tedlar bag)	76	75-125	0-25	1.5	5.0
Ethane(TCD)	EPA 18 (Tedlar bag)	76	75-125	0-25	150	610
Ethane (FID)	EPA 18 (Tedlar bag)	76	75-125	0-25	1.5	5.0
Ethene (TCD)	EPA 18 (Tedlar bag)	76	75-125	0-25	150	670
Ethene (FID)	EPA 18 (Tedlar bag)	76	75-125	0-25	1.5	5.0
Propane (FID)	EPA 18 (Tedlar bag)	76	75-125	0-25	0.37	5.0
Butane(FID)	EPA 18 (Tedlar bag)	76	75-125	0-25	0.84	5.0
Pentane(FID)	EPA 18 (Tedlar bag)	76	75-125	0-25	0.95	5.0
Hexane(FID)	EPA 18 (Tedlar bag)	76	75-125	0-25	1.1	5.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug)	RLA (ug)
Hydrocarbons/Aaromatics						
Cyclohexane	NIOSH 1500/OSHA 07	98/99	75-125	0-25	2.5	10
Cyclohexene	NIOSH 1500/OSHA 07	98/99	75-125	0-25	2.5	10
n-Heptane	NIOSH 1500/OSHA 07	98/99	75-125	0-25	2.5	10
Methylcyclohexane	NIOSH 1500/OSHA 07	98/99	75-125	0-25	2.5	10
n-Octane	NIOSH 1500/OSHA 07	98/99	75-125	0-25	2.5	10
n-Pentane	NIOSH 1500/OSHA 07	98/99	75-125	0-25	2.5	10
Benzene	NIOSH 1500/1501 OSHA 07	98/99	75-125	0-25	2.5	10
Toluene	NIOSH 1500/1501 OSHA 07	98/99	75-125	0-25	2.5	10
Ethylbenzene	NIOSH 1501/OSHA 07	98/99	75-125	0-25	2.5	10
Xylene (total)	NIOSH 1501/OSHA 07	98/99	75-125	0-25	2.5	10
Styrene	NIOSH 1501/OSHA 09	98/99	75-125	0-25	2.5	10
Cumene	NIOSH 1501	98	75-125	0-25	2.5	10
Naphthalene	NIOSH 1501/OSHA 07	98/99	75-125	0-25	2.5	10
a-Methylstyrene	NIOSH 1501/OSHA 07	98/99	75-125	0-25	2.5	10
Vinyltoluene	NISOH 1501/OSHA 07	98/99	75-125	0-25	2.5	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug)	RLA (ug)
Halogenated Hydrocarbons						
Benzyl chloride	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
Bromoform	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
Carbon tetrachloride	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
Chlorobenzene	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
Chlorobromomethane	NIOSH 1003	98	75-125	0-25	2.5	10
Chloroform	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
1,2-Dichlorobenzene	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
1,4,-Dichlorobenzene	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
1,3-Dichlorobenzene	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
1,1,-Dichloroethane	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
1,2-Dichloroethane	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
cis-1,2-Dichloroethene	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
trans-1,2-Dichloroethene	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
Hexachloroethane	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
1,1,1-Trichloroethane	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
Tetrachloroethene	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
1,1,2-Trichloroethane	NIOSH 1003/OSHA 07	98/99	75-125	0-25	2.5	10
1,2,3-Trichloropropane	NIOSH 1003/OSHA 07	98	75-125	0-25	2.5	10
Methylene chloride	NISOH 1005	98	75-125	0-25	2.5	10
Vinyl chloride	NIOSH 1007	98	75-125	0-25	2.5	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Methanol (Methyl alcohol)	NIOSH 2000	98	75-125	0-25	2.5	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Biphenyl (Diphenyl) and Biphenyl oxide	NIOSH 2530	98	75-125	0-25	2.5	10
Acenapthene	NIOSH 5515	98	75-125-	0-25	0.84	10
Acenapthylene	NIOSH 5515	98	75-125	0-25	0.83	10
Anthracene and Phenanthrene	NIOSH 5515	98	75-125	0-25	0.84	10
Benzo(a)anthracene and Chrysene	NIOSH 5515V	98	75-125	0-25	2.4	10
Benzo(a)pyrene	NIOSH 5515	98	75-125	0-25	0.88	10
Benzo (b) fluoranthene and Benzo(k) fluoranthene	NIOSH 5515	98	75-125	0-25	2.6	10
Benzo(g,h,i)perylene	NIOSH 5515	98	75-125	0-25	0.74	10
Fluoranthene	NIOSH 5515	98	75-125	0-25	0.85	10
Fluorene	NIOSH 5515	98	75-125	0-25	0.87	10
Indeno(1,2,3-cd) pyrene and Dibenz(a,h) anthracene	NIOSH 5515	98	75-125	0-25	0.79	10
Naphthalene	NIOSH 5515	98	75-125	0-25	0.78	10
Pyrene	NIOSH 5515	98	75-125	0-25	1.2	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Aluminum (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	3.2	20
Antimony (ICP)	OSHA ID-125G	99	75-125	0-20	0.42	2.0
Antimony (GFAA)	OSHA ID 121	99	75-125	0-20	0.11	1.0
Arsenic (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.39	1.0
Beryllium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.11	0.50
Cadmium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.31	0.50
Cadmium (GFAA)	OSHA ID-121	99	80-120	0-20	0.0090	0.10
Calcium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	2.4	50
Chromium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.44	1.0
Chromium (GFAA)	OSHA ID-121	99	80-120	0-20	0.050	1.0
Cobalt (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.41	1.0
Copper (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.78	2.5
Iron (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	3.9	5.0
Lead (ICP)	NIOSH 7300/OSHA ID-125G	98/2	75-125	0-20	1.0	5.0
·ad (GFAA)	OSHA ID-121	99	80-120	0-20	0.14	0.50
agnesium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	1.8	50
Manganese (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.18	1.0
Mercury (CVAA)	NIOSH 6009	98	75-125	0-20	0.0020	0.0020
Molybdenum (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.23	1.0
Nickel (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	1.1	4.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/)	RLA (ug/)
Selenium(ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.46	1.0
Selenium (GFAA)	OSHA ID-121	99	80-120	0-20	0.13	1.0
Silver (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.20	1.0
Silver (GFAA)	OSHA ID-121	99	80-120	0-20	0.016	0.10
Sodium (ICP)	NIOSH 7300	98	75-125	0-20	8.3	50
Thallium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.46	1.0
Thallium (GFAA)	OSHA ID-121	99	80-120	0-20	0.14	1.0
Tin (ICP)	OSHA ID-125G	99	75-125	0-20	1.3	5.0
Titanium(ICP)	NIOSH 7300	98	70-130	0-30	0.25	1.0
Vanadium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.23	1.0
Zinc (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	0-20	0.45	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Chloride	NIOSH 7903	98	75-125	0-25	2.5	10
Fluoride	NIOSH 7903	98	75-125	0-25	0.25	1.0
Nitrate	NIOSH 7903	98	75-125	0-25	0.25	1.0
Sulfate	NIOSH 7903	98	75-125	0-25	0.13	50

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Aluminum (ICP)	6010(3050)	2	75-125	0-20	3.2	20
Antimony (ICP)	6010(3050)	2	75-125	0-20	0.42	5.0
Antimony (GFAA)	7041(3050)	2	80-120	0-20	0.11	1.0
Arsenic (ICP)	6010(3050)	2	75-125	0-20	0.39	1.0
Arsenic (GFAA)	7060(3050)	2	80-120	0-20	0.27	1.0
Barium (ICP)	6010(3050)	2	75-125	0-20	0.42	1.0
Beryllium (ICP)	6010(3050)	2	75-125	0-20	0.11	0.40
Boron(ICP)	6010(3050)	2	75-125	0-20	0.59	5.0
Cadmium (ICP)	6010(3050)	2	75-125	0-20	0.056	0.50
Cadmium (GFAA)	7131 (3050)	2	80-120	0-20	0.0090	0.10
Calcium (ICP)	6010(3050)	2	75-125	0-20	2.4	50
Chromium (ICP)	6010(3050)	2	75-125	0-20	0.18	1.0
Chromium (GFAA)	7191(3050)	2	80-120	0-20	0.050	1.0
Cobalt (ICP)	6010 (3050)	2	75-125	0-20	0.13	1.0
Copper (ICP)	6010 (3050)	2	75-125	0-20	0.22	2.0
Iron (ICP)	6010 (3050)	2	75-125	0-20	3.9	5.0
Lead (ICP)	6010 (3050)	2	75-125	0-20	0.20	0.50
Lead (GFAA)	7421(3050)	2	80-120	0-20	0.14	0.50
Magnesium (ICP)	6010 (3050)	2	75-125	0-20	1.8	50
Manganese (ICP)	6010(3050)	2	75-125	0-20	0.18	1.0
Mercury (CVAA)	7471	2	80-120	0-20	0.0020	0.020
Molybdenum (ICP)	6010(3050)	2	75-125	0-20	0.098	1.0
Nickel (ICP)	6010(3050)	2	75-125	0-20	1.1	4.0
Potassium (ICP)	6010(3050)	2	75-125	0-20	35	100
Selenium (ICP)	6010 (3050)	2	75-125	0-20	0.46	1.0
Selenium (GFAA)	7740(3050)	2	80-120	0-20	0.13	1.0

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Silver (ICP)	6010 (3050)	2	75-125	0-20	0.14	1.0
Silver (GFAA)	7761 (3050)	2	80-120	0-20	0.016	0.10
Sodium (ICP)	6010(3050)	2	75-125	0-20	8.3	50
Strontium(ICP)	6010(3050)	2	70-130	0-30	0.84	1.0
Thallium (ICP)	6010 (3050)	2	75-125	0-20	0.46	1.0
Thallium (GFAA)	7841 (3050)	2	80-120	0-20	0.14	1.0
Tin (ICP)	6010(3050)	2	75-125	0-20	1.3	5.0
Titanium(ICP)	6010(3050)	2	70-130	0-30	0.25	1.0
Vanadium (ICP)	6010 (3050)	2	75-125	0-20	0.23	1.0
Zinc (ICP)	6010 (3050)	2	75-125	0-20	0.45	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer
-T = trace ICP

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Cyanide, total	9012(9013)	2	75-125	0-30	0.59	1.0
	9012(9010)	2	75-125	0-30	0.14	1.0

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Aldrin (MS)	8081(3550)	97/2	10-144	0-38	0.021	1.7
Alpha-BHC	8081(3550)	97/2	22-101	0-40	0.088	1.7
Beta-BHC	8081(3550)	97/2	12-120	0-40	0.16	1.7
Gamma-BHC (Lindane) (MS)	8081(3550)	97/2	12-138	0-37	0.066	1.7
Delta-BHC	8081(3550)	97/2	10-142	0-47	0.18	1.7
Technical Chlordane	8081(3550)	97/2	45-119	0-40	1.7	17
Alpha Chlordane	8081(3550)	97/2	45-140	0-40	0.12	1.7
Gamma Chlordane	8081(3550)	97/2	11-141	0-40	0.078	1.7
4,4'-DDD	8081(3550)	97/2	28-134	0-50	0.22	3.3
4,4'-DDE	8081(3550)	97/2	34-121	0-23	0.13	3.3
4,4'-DDT (MS)	8081(3550)	97/2	29-134	0-26	0.49	3.3
Dieldrin (MS)	8081(3550)	97/2	28-137	0-30	0.19	3.3
Endosulfan I	8081(3550)	97/2	10-141	0-40	0.13	1.7
Endosulfan II	8081(3550)	97/2	10-159	0-65	0.22	3.3
ndosulfan sulfate	8081(3550)	97/2	26-144	0-50	0.91	3.3
Endrin (MS)	8081(3550)	97/2	33-149	0-32	0.10	3.3
Endrin aldehyde	8081(3550)	97/2	10-130	0-86	0.22	3.3
Endrin ketone	8081(3550)	97/2	29-112	0-31	0.42	3.3
Heptachlor (MS)	8081(3550)	97/2	17-138	0-38	0.12	1.7
Heptachlor epoxide	8081(3550)	97/2	15-142	0-40	0.099	1.7
Methoxychlor	8081(3550)	97/2	24-152	0-40	1.3	17
Toxaphene	8081(3550)	97/2	41-126	0-50	18	170
PCB-1016	8082(3550)	97/2	34-137	0-44	4.8	33
PCB 1221	8082(3550)	97/2	15-178	0-30	23	67
PCB 1232	8082(3550)	97/2	10-215	0-30	4.0	33
PCB-1242	8082(3550)	97/2	39-150	0-30	6.0	33
PCB-1248	8082(3550)	97/2	38-158	0-30	6.7	33
PCB-1254	8082(3550)	97/2	66-122	0-30	2.7	33
PCB-1260	8082(3550)	97/2	58-150	0-30	5.6	33
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081(3550)/8082(3550)	97/2	10-114	NA	NA	NA
Surrogate-Decachlorobiphenyl (DCB)	8081(3550)/8082(3550)	97/2	27-128	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Monochlorobiphenyls	680/(3550)	97/93	30-130	0-50	0.68	10
Dihlorobiphenyls	680/(3550)	97/93	30-130	0-50	0.76	10
Trichlorobiphenyls	680/(3550)	97/93	30-130	0-50	0.68	10
Tetrachlorobiphenyls	680/(3550)	97/93	40-140	0-50	1.3	20
Pentachlorobiphenyls	680/(3550)	97/93	40-140	0-50	0.83	20
Hexachlorobiphenyls	680/(3550)	97/93	40-140	0-50	0.89	20
Heptachlorobiphenyls	680/(3550)	97/93	40-140	0-50	1.6	30
Octachlorobiphenyls	680/(3550)	97/93	40-140	0-50	0.95	30
Nonachlorobiphenyls	680/(3550)	97/93	30-130	0-50	1.9	50
Decachlorobiphenyl	680/(3550)	97/93	30-130	0-50	1.9	50
Surrogate- Decachlorobiphenyl-13C12	680/(3550)	97/93	30-130	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acetone	8260(5035)	97/2	14-189	0-40	2.3	50
	8260(5035ext)	97/2	14-189	0-40	230	5000
Benzene (MS)	8260(5035)	97/2	64-144	0-25	0.41	5.0
	8260(5035ext)	97/2	64-144	0-25	41	500
Bromodichloromethane	8260(5035)	97/2	71-140	0-40	0.34	5.0
	8260(5035ext)	97/2	71-140	0-40	34	500
Bromoform	8260(5035)	97/2	59-143	0-40	0.44	5.0
	8260(5035ext)	97/2	59-143	0-40	44	500
Bromomethane (Methyl bromide)	8260(5035)	97/2	35-181	0-65	2.0	10
	8260(5035ext)	97/2	35-181	0-65	200	1000
2-Butanone (MEK)	8260(5035)	97/2	54-166	0-40	2.8	25
	8260(5035ext)	97/2	54-166	0-40	280	2500
Carbon disulfide	8260(5035)	97/2	10-160	0-65	0.29	5.0
	8260(5035ext)	97/2	10-160	0-65	29	500
arbon tetrachloride	8260(5035)	97/2	65-129	0-40	0.47	5.0
	8260(5035ext)	97/2	65-129	0-40	47	500
Chlorobenzene (MS)	8260(5035)	97/2	56-152	0-25	0.58	5.0
	8260(5035ext)	97/2	56-152	0-25	58	500
Chloroethane	8260(5035)	97/2	65-129	0-40	1.3	10
	8260(5035ext)	97/2	65-129	0-40	130	1000
Chloroform	8260(5035)	97/2	62-117	0-40	0.26	5.0
	8260(5035ext)	97/2	62-117	0-40	26	500
Chloromethane	8260(5035)	97/2	10-186	0-65	1.2	10
	8260(5035ext)	97/2	10-186	0-65	120	1000
Dibromochloromethane	8260(5035)	97/2	73-127	0-40	0.28	5.0
	8260(5035ext)	97/2	73-127	0-40	28	5.0
1,1-Dichloroethane	8260(5035)	97/2	64-112	0-40	0.28	5.0
	8260(5035ext)	97/2	64-112	0-40	28	500
1,2-Dichloroethane	8260(5035)	97/2	66-128	0-40	1.0	5.0
	8260(5035ext)	97/2	66-128	0-40	100	500
cis-1,2-Dichloroethene	8260(5035)	97/2	64-123	0-40	0.31	5.0
	8260(5035ext)	97/2	64-123	0-40	31	500
trans-1,2-Dichloroethene	8260(5035)	97/2	60-161	0-25	0.57	5.0
	8260(5035ext)	97/2	60-161	0-25	57	500
1,1-Dichloroethene (MS)	8260(5035)	97/2	44-157	0-25	0.77	5.0
	8260(5035ext)	97/2	44-157	0-25	77	500
1,2-Dichloropropane	8260(5035)	97/2	47-146	0-65	1.0	5.0
	8260(5035ext)	97/2	47-146	0-65	100	500

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
cis-1,3-Dichloropropene	8260(5035)	97/2	50-146	0-65	0.49	5.0
	8260(5035ext)	97/2	50-146	0-65	49	500
trans-1,3-Dichloropropene	8260(5035)	97/2	57-143	0-65	0.65	5.0
	8260(5035ext)	97/2	57-143	0-65	65	500
Ethylbenzene	8260(5035)	97/2	60-131	0-40	0.55	5.0
	8260(5035ext)	97/2	60-131	0-40	55	500
2-Hexanone	8260(5035)	97/2	47-145	0-40	3.7	25
	8260(5035ext)	97/2	47-145	0-40	370	2500
4-Methyl-2-pentanone (MIBK)	8260(5035)	97/2	54-167	0-49	3.2	25
	8260(5035ext)	97/2	54-167	0-49	320	2500
Styrene	8260(5035)	97/2	56-121	0-40	0.43	5.0
	8260(5035ext)	97/2	56-121	0-40	.43	500
1,1,2,2-Tetrachloroethane	8260(5035)	97/2	36-157	0-40	0.80	5.0
	8260(5035ext)	97/2	36-157	0-40	80	500
Tetrachloroethene	8260(5035)	97/2	61-142	0-40	0.44	5.0
	8260(5035ext)	97/2	61-142	0-40	44	500
Toluene (MS)	8260(5035)	97/2	67-142	0-25	0.48	5.0
	8260(5035ext)	97/2	67-142	0-25	48	500
1,1,1-Trichloroethane	8260(5035)	97/2	11-148	0-40	0.98	5.0
	8260(5035ext)	97/2	11-148	0-40	98	500
1,1,2-Trichloroethane	8260(5035)	97/2	66-123	0-40	0.65	5.0
	8260(5035ext)	97/2	66-123	0-40	65	500

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Trichloroethene (MS)	8260(5035)	97/2	41-134	0-22	0.72	5.0
	8260(5035ext)	97/2	41-134	0-22	72	500
Vinyl chloride	8260(5035)	97/2	18-169	0-65	0.98	10
	8260(5035ext)	97/2	18-169	0-65	98	1000
Xylenes (total)	8260(5035)	97/2	50-150	0-40	1.5	5.0
	8260(5035ext)	97/2	50-150	0-40	150	500
o-Xylene	8260(5035)	97/2	22-154	0-40	0.50	5.0
	8260(5035ext)	97/2	22-154	0-40	50	500
m+p-Xylene	8260(5035)	97/2	62-123	0-40	1.0	5.0
	8260(5035ext)	97/2	62-123	0-40	100	500
Surrogate - Toluene-d8	8260(5035)	97/2	58-148	NA	NA	NA
	8260(5035ext)	97/2	58-148	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5035)	97/2	70-136	NA	NA	NA
	8260(5035ext)	97/2	70-136	NA	NA	NA
Surrogate -Dibromofluoromethane	8260(5035)	97/2	66-148	NA	NA	NA
	8260(5035ext)	97/2	66-148	NA	NA	NA
rogate - 1,2-Dichloroethane-d4	8260(5035)	97/2	38-151	NA	NA	NA
	8260(5035ext)	97/2	38-151	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	8260(5035)	97/2	58-148	NA	NA	NA
	8260(5035ext)	97/2	58-148	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acenaphthene (MS)	8270(3550)	97/2	28-102	0-25	13	330
Acenaphthylene	8270(3550)	97/2	54-140	0-25	13	330
Anthracene	8270(3550)	97/2	39-106	0-30	12	330
Benzo(a)anthracene	8270(3550)	97/2	29-111	0-25	9.6	330
Benzoic acid	8270(3550)	97/2	10-150	0-50	21	1700
Benzo(b)fluoranthene	8270(3550)	97/2	19-126	0-25	17	330
Benzo(k)fluoranthene	8270(3550)	97/2	26-125	0-38	13	330
Benzo(g,h,i)perylene	8270(3550)	97/2	10-121	0-50	19	330
Benzo(a)pyrene	8270(3550)	97/2	34-116	0-29	11	330
Bis(2-chloroethoxy) methane	8270(3550)	97/2	13-108	0-50	30	330
Bis(2-chloroethyl) ether	8270(3550)	97/2	10-130	0-50	9.6	330
Bis(2-chloroisopropyl) ether	8270(3550)	97/2	36-166	0-50	9.9	330
Bis(2-ethylhexyl) phthalate	8270(3550)	97/2	23-124	0-40	46	330
4-Bromophenyl phenyl ether	8270(3550)	97/2	53-127	0-40	11	330
Butyl benzyl phthalate	8270(3550)	97/2	24-120	0-40	30	330
4-Chloroaniline	8270(3550)	97/2	10-150	0-50	17	660
4-Chloro-3-methylphenol (MS) (p-Chloro-m-cresol)	8270(3550)	97/2	25-107	0-25	10	330
2-Chloronaphthalene	8270(3550)	97/2	60-118	0-40	11	330
2-Chlorophenol (MS)	8270(3550)	97/2	23-114	0-25	11	330
4-Chlorophenylphenyl ether	8270(3550)	97/2	20-118	0-33	10	330
Chrysene	8270(3550)	97/2	35-130	0-27	12	330
2-Methyl phenol (o-Cresol)	8270(3550)	97/2	14-95	0-50	13	330
3- Methyl phenol (m- Cresol)	8270(3550)	97/2	12-102	0-50	61	330
4-Methyl phenol (p-Cresol)	8270(3550)	97/2	12-102	0-50	61	330
3- and 4-Methyl phenol (m- and p-Cresol)	8270(3550)	97/2	12-102	0-50	61	330
Dibenz(a,h)anthracene	8270(3550)	97/2	40-147	0-28	15	330
Dibenzofuran	8270(3550)	97/2	10-150	0-50	12	330
Di-n-butylphthalate	8270(3550)	97/2	28-125	0-50	15	330
1,2-Dichlorobenzene	8270(3550)	97/2	10-99	0-40	11	330
1,3-Dichlorobenzene	8270(3550)	97/2	10-100	0-42	8.8	330
1,4-Dichlorobenzene (MS)	8270(3550)	97/2	10-125	0-40	11	330
3,3'-Dichlorobenzidine	8270(3550)	97/2	10-189	0-100	16	660
2,4-Dichlorophenol	8270(3550)	97/2	16-104	0-40	10	330
Diethylphthalate	8270(3550)	97/2	31-113	0-40	15	330
2,4-Dimethylphenol	8270(3550)	97/2	10-95	0-25	10	330
Dimethylphthalate	8270(3550)	97/2	20-116	0-40	11	330
4,6-Dinitro-2-methylphenol	8270(3550)	97/2	43-135	0-93	29	1700
2,4-Dinitrophenol	8270(3550)	97/2	10-126	0-87	22	1700

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,4-Dinitrotoluene (MS)	8270(3550)	97/2	26-107	0-30	13	330
2,6-Dinitrotoluene	8270(3550)	97/2	38-107	0-40	16	330
Di-n-octylphthalate	8270(3550)	97/2	22-135	0-50	19	330
Fluoranthene	8270(3550)	97/2	23-121	0-25	11	330
Fluorene	8270(3550)	97/2	32-107	0-40	12	330
Hexachlorobenzene	8270(3550)	97/2	44-105	0-40	10	330
Hexachlorobutadiene	8270(3550)	97/2	10-112	0-40	11	330
Hexachlorocyclopentadiene	8270(3550)	97/2	D-132	0-50	18	330
Hexachloroethane	8270(3550)	97/2	16-91	0-40	13	330
Indeno(1,2,3-cd)pyrene	8270(3550)	97/2	18-157	0-83	20	330
Isophorone	8270(3550)	97/2	17-107	0-60	12	330
2-Methylnaphthalene	8270(3550)	97/2	19-105	0-50	9.2	330
Naphthalene	8270(3550)	97/2	53-125	0-25	11	330
2-Nitroaniline	8270(3550)	97/2	22-110	0-50	14	1700
Nitroaniline	8270(3550)	97/2	10-117	0-50	11	1700
Nitroaniline	8270(3550)	97/2	10-136	0-50	13	1700
Nitrobenzene	8270(3550)	97/2	9-104	0-40	13	330
2-Nitrophenol	8270(3550)	97/2	10-101	0-40	8.8	330
4-Nitrophenol (MS)	8270(3550)	97/2	10-117	0-45	30	1700
N-Nitrosodiphenylamine/ Diphenylamine	8270(3550)	97/2	40-122	0-50	98	330
N-Nitroso-di-n-propylamine MS)	8270(3550)	97/2	11-117	0-35	14	330
Pentachlorophenol (MS)	8270(3550)	97/2	10-120	0-44	28	1700
Phenanthrene	8270(3550)	97/2	38-113	0-25	12	330
Phenol (MS)	8270(3550)	97/2	17-103	0-25	19	330
Pyrene (MS)	8270(3550)	97/2	18-136	0-25	11	330
1,2,4-Trichlorobenzene (MS)	8270(3550)	97/2	17-105	0-28	11	330
2,4,5-Trichlorophenol	8270(3550)	97/2	39-123	0-27	13	330
2,4,6-Trichlorophenol	8270(3550)	97/2	20-116	0-40	9.2	330
Surrogate - Nitrobenzene-d5	8270(3550)	97/2	12-125	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	8270(3550)	97/2	24-118	NA	NA	NA
Surrogate - p-Terphenyl-d14	8270(3550)	97/2	18-153	NA	NA	NA
Surrogate - Phenol-d5	8270(3550)	97/2	10-142	NA	NA	NA
Surrogate - 2-Fluorophenol	8270(3550)	97/2	10-118	NA	NA	NA
Surrogate - 2,4,6-Tribromophenol	8270(3550)	97/2	14-121	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD)	8280	97/2	69-145	0-40	0.13	0.50
Polychlorinated Dibenzo-p-dioxins and Dibenzofurans classes						
tetra-CDD	8280	97/2	69-145	0-40	0.12	0.50
tetra-CDF	8280	97/2	59-142	0-40	0.040	0.50
penta-CDD	8280	97/2	41-203	0-40	0.089	0.50
penta-CDF	8280	97/2	55-146	0-40	0.068	0.50
hexa-CDD	8280	97/2	45-174	0-53	0.13	0.50
hexa-CDF	8280	97/2	50-154	0-46	0.051	0.50
hepta-CDD	8280	97/2	20-170	0-50	0.13	1.0
hepta-CDF	8280	97/2	20-170	0-50	0:063	1.0
octa-CDD	8280	97/2	20-170	0-50	0.14	1.0
octa-CDF	8280	97/2	20-170	0-50	0.13	1.0
Internal Standard - 13C12-2,3,7,8-TCDD	8280	97/2	25-150	NA	NA	NA
Internal Standard - 13C12-2,3,7,8-TCDF	8280	97/2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,6,7,8-HxCDD	8280	97/2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,4,6,7,8-HpCDF	8280	97/2	25-150	NA	NA	NA
Internal Standard - 13C12-OCDD	8280	97/2	25-150	NA	NA	NA

TABLE 5.5. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acenaphthene (MS)	8310(3550)	97/2	11-144	0-35	14	50
Acenaphthylene	8310(3550)	97/2	10-139	0-40	5.1	20
Anthracene	8310(3550)	97/2	10-126	0-40	0.23	4.0
Benzo(a)anthracene	8310(3550)	97/2	12-135	0-40	0.27	4.0
Benzo(b)fluoranthene	8310(3550)	97/2	10-150	0-40	0.15	4.0
Benzo(k)fluoranthene	8310(3550)	97/2	10-159	0-40	0.12	4.0
Benzo(g,h,i)perylene	8310(3550)	97/2	10-120	0-40	0.68	10
Benzo(a)pyrene	8310(3550)	97/2	10-128	0-40	0.35	4.0
Chrysene (MS)	8310(3550)	97/2	10-199	0-40	0.28	4.0
Dibenzo(a,h)anthracene	8310(3550)	97/2	10-110	0-40	0.87	10
Fluoranthene	8310(3550)	97/2	56-136	0-28	0.47	10
luorene (MS)	8310(3550)	97/2	10-142	0-40	1.4	10
Indeno(1,2,3-cd)pyrene	8310(3550)	97/2	10-116	0-40	0.25	10
1-Methylnaphthalene	8310(3550)	97/2	10-125	0-40	6.2	20
2-Methylnaphthalene	8310(3550)	97/2	10-125	0-40	5.9	20
Naphthalene (MS)	8310(3550)	97/2	31-159	0-34	5.3	20
Phenanthrene	8310(3550)	97/2	10-155	0-40	0.50	4.0
Pyrene (MS)	8310(3550)	97/2	49-156	0-28	0.93	10
Surrogate - 4-Terphenyl-d14	8310(3550)	97/2	28-151	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Aluminum (ICP)	6010(3050)	98/2	75-125	0-20	3.2	20
Antimony (ICP)	6010(3050)	98/2	75-125	0-20	0.42	2.0
Antimony (GFAA)	7041(3050)	98/2	80-120	0-20	0.11	1.0
Arsenic (ICP)	6010(3050)	98/2	75-125	0-20	0.39	1.0
Arsenic (GFAA)	7060(3050)	98/2	80-120	0-20	0.27	1.0
Barium (ICP)	6010(3050)	98/2	75-125	0-20	0.42	1.0
Beryllium (ICP)	6010(3050)	98/2	75-125	0-20	0.11	0.40
Boron	6010(3050)	98/2	75-125	0-20	0.59	5.0
Cadmium (ICP)	6010(3050)	98/2	75-125	0-20	0.056	0.50
Cadmium (GFAA)	7131 (3050)	98/2	80-120	0-20	0.0090	0.10
Calcium (ICP)	6010(3050)	98/2	75-125	0-20	2.4	50
Chromium (ICP)	6010(3050)	98/2	75-125	0-20	0.18	1.0
Chromium (GFAA)	7191(3050)	98/2	80-120	0-20	0.050	1.0
Cobalt (ICP)	6010 (3050)	98/2	75-125	0-20	0.13	1.0
Copper (ICP)	6010 (3050)	98/2	75-125	0-20	0.22	2.0
Iron (ICP)	6010 (3050)	98/2	75-125	0-20	3.9	5.0
Lead (ICP)	6010 (3050)	98/2	75-125	0-20	0.20	0.50
Lead (GFAA)	7421(3050)	98/2	80-120	0-20	0.14	0.50
Magnesium (ICP)	6010 (3050)	98/2	75-125	0-20	1.8	50
Manganese (ICP)	6010(3050)	98/2	75-125	0-20	0.18	1.0
Mercury (CVAA)	7471	98/2	80-120	0-20	0.0020	0.020
Molybdenum (ICP)	6010(3050)	98/2	75-125	0-20	0.098	1.0
Nickel (ICP)	6010(3050)	98/2	75-125	0-20	1.1	4.0
Potassium (ICP)	6010(3050)	98/2	75-125	0-20	35	100
Selenium (ICP)	6010 (3050)	98/2	75-125	0-20	0.46	1.0
Selenium (GFAA)	7740(3050)	98/2	80-120	0-20	0.13	1.0

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Silver (ICP)	6010 (3050)	98/2	75-125	0-20	0.14	1.0
Silver (GFAA)	7761 (3050)	98/2	80-120	0-20	0.016	0.10
Sodium (ICP)	6010(3050)	98/2	75-125	0-20	8.3	50
Strontium(ICP)	6010(3050)	98/2	75-125	0-30	0.84	1.0
Thallium (ICP)	6010 (3050)	98/2	75-125	0-20	0.46	1.0
Thallium (GFAA)	7841 (3050)	98/2	80-120	0-20	0.14	1.0
Tin (ICP)	6010(3050)	98/2	75-125	0-20	1.3	5.0
Titanium(ICP)	6010(3050)	98/2	70-130	0-30	0.25	1.0
Vanadium (ICP)	6010 (3050)	98/2	75-125	0-20	0.23	1.0
Zinc (ICP)	6010 (3050)	98/2	75-125	0-20	0.45	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL* (ug/wipe)	RLA (ug/wipe)
Aldrin (MS)	8081(3550)	98/2	10-144	0-38	0.0041	0.050
alpha-BHC	8081(3550)	98/2	22-101	0-40	0.0022	0.050
beta-BHC	8081(3550)	98/2	12-120	0-40	0.0053	0.050
gamma-BHC (Lindane) (MS)	8081(3550)	98/2	12-138	0-37	0.00119	0.050
delta-BHC	8081(3550)	98/2	10-142	0-47	0.0019	0.050
technical Chlordane	8081(3550)	98/2	45-119	0-40	0.061	0.20
alpha Chlordane	8081(3550)	98/2	45-140	0-40	0.0028	0.050
gamma Chlordane	8081(3550)	98/2	11-141	0-40	0.0026	0.050
4,4'-DDD	8081(3550)	98/2	28-134	0-50	0.0060	0.10
4,4'-DDE	8081(3550)	98/2	34-121	0-25	0.0069	0.10
4,4'-DDT (MS)	8081(3550)	98/2	29-134	0-26	0.0074	0.10
Dieldrin (MS)	8081(3550)	98/2	28-137	0-30	0.0023	0.10
Endosulfan I	8081(3550)	98/2	10-141	0-40	0.0027	0.050
Endosulfan II	8081(3550)	98/2	10-159	0-65	0.0037	0.10
Endosulfan sulfate	8081(3550)	98/2	26-144	0-50	0.0053	0.10
Endrin (MS)	8081(3550)	98/2	33-149	0-32	0.0085	0.10
Endrin aldehyde	8081(3550)	98/2	10-130	0-86	0.0079	0.10
Endrin ketone	8081(3550)	98/2	29-112	0-31	0.011	0.10
Heptachlor (MS)	8081(3550)	98/2	17-138	0-38	0.0025	0.050
Heptachlor epoxide	8081(3550)	98/2	15-142	0-40	0.0085	0.050
Methoxychlor	8081(3550)	98/2	24-152	0-40	0.032	0.50
Toxaphene	8081(3550)	98/2	41-126	0-50	0.55	5.0
PCB-1016	8082(3550)	98/2	34-137	0-44	0.15	1.0
PCB 1221	8082(3550)	98/2	15-178	0-30	0.27	2.0
PCB 1232	8082(3550)	98/2	10-215	0-30	0.21	1.0
PCB-1242	8082(3550)	98/2	39-150	0-30	0.25	1.0
PCB-1248	8082(3550)	98/2	38-158	0-30	0.18	1.0
PCB-1254	8082(3550)	98/2	66-122	0-30	0.11	1.0
PCB-1260	8082(3550)	98/2	58-150	0-30	0.12	1.0
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081(3550)/ 8082(3550)	98/2	10-114	NA	NA	NA
Surrogate-Decachlorobiphenyl (DCB)	8081(3550)/ 8082(3550)	98/2	27-128	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Monochlorobiphenyls	680/(3550)	98/93	30-130	0-50	0.044	0.30
Dihlorobiphenyls	680/(3550)	98/93	30-130	0-50	0.035	0.30
Trichlorobiphenyls	680/(3550)	98/93	30-130	0-50	0.035	0.30
Tetrachlorobiphenyls	680/(3550)	98/93	40--140	0-50	0.053	0.60
Pentachlorobiphenyls	680/(3550)	98/93	40--140	0-50	0.029	0.60
Hexachlorobiphenyls	680/(3550)	98/93	40--140	0-50	0.037	0.60
Heptachlorobiphenyls	680/(3550)	98/93	40--140	0-50	0.042	0.90
Octachlorobiphenyls	680/(3550)	98/93	40--140	0-50	0.064	0.90
Nonachlorobiphenyls	680/(3550)	98/93	30-130	0-50	0.11	1.5
Decachlorobiphenyl	680/(3550)	98/93	30-130	0-50	0.11	1.5
Surrogate- Decachlorobiphenyl-13C12	680/(3550)	98/93	30-130	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Acephate(1)	1657	98/72	25-140	0-50	1.2	5.0
Methamidophos(1)	1657	98/72	36-106	0-46	0.10	2.0
Alachlor	8141(3520)	98/2	46-139	0-30	0.14	1.0
Ametryn	8141(3520)	98/2	60-120	0-40	0.16	2.0
Atrazine (MS)	8141(3520)	98/2	39-130	0-30	0.13	2.0
Azinphos methyl	8141(3520)	98/2	48-162	0-50	0.16	1.0
Benoxacor	8141(3520)	98/2	10-150	0-50	4.4	10
Bolstar	8141(3520)	98/2	36-114	0-40	0.26	1.0
Butachlor	8141(3520)	98/2	50-150	0-40	0.35	1.0
Carbophenothion	8141(3520)	98/2	69-122	0-40	0.23	1.0
Chlordimeform (Galecron)	8141(3520)	98/2	10-150	0-50	3.8	10
5-Chloroaminotoluene	8141(3520)	98/2	10-150	0-50	3.4	10
Chlorpyrifos	8141(3520)	98/2	49-109	0-40	0.17	1.0
Chlorpyrifos methyl	8141(3520)	98/2	53-136	0-40	0.17	1.0
Coumaphos	8141(3520)	98/2	61-139	0-40	0.19	1.0
Demeton-o	8141(3520)	98/2	10-117	0-40	1.1	2.5
Demeton-s	8141(3520)	98/2	37-121	0-40	0.35	2.5
Demeton-o+s	8141(3520)	98/2	10-117	0-40	1.1	2.5
Diazinon (MS)	8141(3520)	98/2	40-137	0-40	0.17	1.0
Dichlofenthion	8141(3520)	98/2	38-118	0-40	0.11	1.0
Dichlorvos	8141(3520)	98/2	11-158	0-40	0.28	2.0
Dimethoate	8141(3520)	98/2	14-101	0-40	0.11	2.0
Dioxathion	8141(3520)	98/2	26-127	0-40	1.5	10
Disulfoton	8141(3520)	98/2	42-112	0-66	0.17	2.0
EPN	8141(3520)	98/2	48-124	0-40	0.19	1.0
Ethion	8141(3520)	98/2	62-175	0-40	0.12	0.50
Ethoprop	8141(3520)	98/2	42-123	0-40	0.27	0.50
Famphur	8141(3520)	98/2	13-128	0-60	0.25	2.0
Fenamiphos	8141	98/2	40-160	0-40	0.59	2.0
Fensulfothion	8141(3520)	98/2	31-163	0-40	0.27	5.0

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/wipe)	RLA (ug/ wipe)
Fenthion	8141(3520)	98/2	41-115	0-60	0.23	1.0
Isofenphos	8141	98/2	40-160	0-40	0.060	0.50
Malathion	8141(3520)	98/2	10-140	0-40	0.071	1.0
Merphos	8141(3520)	98/2	32-138	0-40	0.22	1.0
Metolachlor	8141(3520)	98/2	53-133	0-40	0.11	1.0
Metrabuzin	8141(3520)	98/2	50-150	0-40	0.11	1.0
Mevinphos	8141(3520)	98/2	24-166	0-40	0.23	2.0
Monocrotophos	8141(3520)	98/2	43-126	0-50	2.9	10
Naled	8141(3520)	98/2	10-119	0-40	0.14	5.0
Parathion, ethyl (MS)	8141(3520)	98/2	28-155	0-34	0.083	1.0
Parathion, methyl (MS)	8141(3520)	98/2	38-149	0-32	0.19	0.50
Phorate	8141(3520)	98/2	28-119	0-40	0.30	1.0
Prometon	8141(3520)	98/2	55-124	0-40	0.11	2.0
Prometryn	8141(3520)	98/2	36-155	0-40	0.23	2.0
Propazine	8141(3520)	98/2	51-127	0-30	0.25	2.0
Ronnel (MS)	8141(3520)	98/2	30-98	0-35	0.26	1.0
Simazine	8141(3520)	98/2	39-149	0-50	0.19	2.0
Stirophos (Tetrachlorvinphos)	8141(3520)	98/2	48-125	0-40	0.39	1.0
Sulfotepp	8141(3520)	98/2	40-157	0-40	0.17	0.50
Terbufos	8141(3520)	98/2	40-160	0-40	0.16	1.0
Terbutylazine	8141(3520)	98/2	60-130	0-40	0.33	2.0
Terbutryn	8141(3520)	98/2	53-113	0-40	0.066	2.0
Thionazin (MS)	8141(3520)	98/2	12-139	0-60	0.21	1.0
Tokuthion (Prothiofos)	8141(3520)	98/2	45-114	0-40	0.22	1.0
Trichloronate	8141(3520)	98/2	16-123	0-40	0.12	1.0
Surrogate - Triphenylphosphate	8141	98/2	16-164	NA	NA	NA

(1) Determined by NPD

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Acetone	8260(5030)	98/2	14-189	0-40	1.7	5.0
Benzene (MS)	8260(5030)	98/2	64-144	0-25	0.17	0.50
Bromodichloromethane	8260(5030)	98/2	71-140	0-40	0.17	0.50
Bromoform	8260(5030)	98/2	59-143	0-40	0.19	0.50
Bromomethane	8260(5030)	98/2	35-181	0-65	0.31	1.0
2-Butanone (MEK)	8260(5030)	98/2	54-166	0-40	2.6	2.5
Carbon disulfide	8260(5030)	98/2	10-160	0-65	0.090	0.50
Carbon tetrachloride	8260(5030)	98/2	65-129	0-40	0.25	0.50
Chlorobenzene (MS)	8260(5030)	98/2	56-152	0-25	0.18	0.50
Chloroethane	8260(5030)	98/2	65-129	0-40	0.65	1.0
Chloroform	8260(5030)	98/2	62-117	0-40	0.20	0.50
Chloromethane	8260(5030)	98/2	10-186	0-65	0.24	1.0
Dibromochloromethane	8260(5030)	98/2	73-127	0-40	0.17	0.50
1,1-Dichloroethane	8260(5030)	98/2	64-112	0-40	0.21	0.50
1,2-Dichloroethane	8260(5030)	98/2	66-128	0-40	0.20	0.50
cis-1,2-Dichloroethene	8260(5030)	98/2	64-123	0-40	0.25	0.50
trans-1,2-Dichloroethene	8260(5030)	98/2	60-161	0-25	0.32	0.50
1,1-Dichloroethene (MS)	8260(5030)	98/2	44-157	0-23	0.30	0.50
1,2-Dichloropropane	8260(5030)	98/2	47-146	0-65	0.21	0.50
cis-1,3-Dichloropropene	8260(5030)	98/2	50-146	0-65	0.18	0.50
trans-1,3-Dichloropropene	8260(5030)	98/2	57-143	0-65	0.18	0.50
Ethylbenzene	8260(5030)	98/2	60-131	0-40	0.18	0.50
2-Hexanone	8260(5030)	98/2	47-145	0-40	2.2	2.5
4-Methyl-2-pentanone (MIBK)	8260(5030)	98/2	54-167	0-49	1.8	2.5
Styrene	8260(5030)	98/2	56-121	0-40	0.20	0.50
1,1,2,2-Tetrachloroethane	8260(5030)	98/2	36-157	0-40	0.26	0.50
Tetrachloroethene	8260(5030)	98/2	61-142	0-40	0.23	0.50

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RL^ (ug/wipe)
Toluene (MS)	8260(5030)	98/2	67-142	0-25	0.16	0.50
1,1,1-Trichloroethane	8260(5030)	98/2	11-148	0-40	0.16	0.50
1,1,2-Trichloroethane	8260(5030)	98/2	66-123	0-40	0.20	0.50
Trichloroethene (MS)	8260(5030)	98/2	41-134	0-22	0.20	0.50
Vinyl chloride	8260(5030)	98/2	18-169	0-65	0.44	1.0
Xylenes (total)	8260(5030)	98/2	50-150	0-40	0.37	0.50
o-Xylene	8260(5030)	98/2	22-154	0-40	0.20	0.50
m+p-Xylene	8260(5030)	98/2	62-123	0-40	0.23	0.50
Surrogate - Toluene-d8	8260(5030)	98/2	58-148	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5030)	98/2	70-136	NA	NA	NA
Surrogate -Dibromofluoromethane	8260(5030)	98/2	66-148	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	8260(5030)	98/2	38-151	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	8260(5030)	98/2	58-148	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Acenaphthene (MS)	8270(3550)	98/2	28-102	0-25	0.71	10
Acenaphthylene	8270(3550)	98/2	54-140	0-25	1.8	10
Anthracene	8270(3550)	98/2	39-106	0-30	0.42	10
Benzo(a)anthracene	8270(3550)	98/2	29-111	0-25	0.59	10
Benzoic acid	8270(3550)	98/2	10-150	0-50	7.4	50
Benzo(b)fluoranthene	8270(3550)	98/2	19-126	0-25	0.64	10
Benzo(k)fluoranthene	8270(3550)	98/2	26-125	0-38	0.085	10
Benzo(g,h,i)perylene	8270(3550)	98/2	10-121	0-50	0.92	10
Benzo(a)pyrene	8270(3550)	98/2	34-116	0-29	0.58	10
Bis(2-chloroethoxy) methane	8270(3550)	98/2	13-108	0-50	1.7	10
Bis(2-chloroethyl) ether	8270(3550)	98/2	10-130	0-50	1.3	10
Bis(2-chloroisopropyl) ether	8270(3550)	98/2	36-166	0-50	1.3	10
Bis(2-ethylhexyl) phthalate	8270(3550)	98/2	23-124	0-40	0.61	10
4-Bromophenyl phenyl ether	8270(3550)	98/2	53-127	0-40	2.0	10
Butyl benzyl phthalate	8270(3550)	98/2	24-120	0-40	2.2	10
4-Chloroaniline	8270(3550)	98/2	10-150	0-50	1.4	20
4-Chloro-3-methylphenol (MS) (p-Chloro-m-cresol)	8270(3550)	98/2	25-107	0-25	0.82	10
2-Chloronaphthalene	8270(3550)	98/2	60-118	0-40	1.8	10
2-Chlorophenol (MS)	8270(3550)	98/2	23-114	0-25	1.4	10
4-Chlorophenylphenyl ether	8270(3550)	98/2	20-118	0-33	2.1	10
Chrysene	8270(3550)	98/2	35-130	0-27	0.46	10
2-Methyl phenol (o-Cresol)	8270(3550)	98/2	14-95	0-50	1.5	10
3- Methyl phenol (m- Cresol)	8270(3550)	98/2	12-102	0-50	1.5	10
4-Methyl phenol (p-Cresol)	8270(3550)	98/2	12-102	0-50	1.5	10
3- and 4-Methyl phenol (m- and p-Cresol)	8270(3550)	98/2	12-102	0-50	1.5	10
Dibenz(a,h)anthracene	8270(3550)	98/2	40-147	0-28	0.83	10
Dibenzofuran	8270(3550)	98/2	10-150	0-50	2.3	10
Di-n-butylphthalate	8270(3550)	98/2	28-125	0-50	0.65	10
1,2-Dichlorobenzene	8270(3550)	98/2	10-99	0-40	1.5	10
1,3-Dichlorobenzene	8270(3550)	98/2	10-100	0-42	1.3	10
1,4-Dichlorobenzene (MS)	8270(3550)	98/2	10-125	0-40	2.3	10
3,3'-Dichlorobenzidine	8270(3550)	98/2	10-189	0-100	8.0	20
2,4-Dichlorophenol	8270(3550)	98/2	16-104	0-40	1.6	10
Diethylphthalate	8270(3550)	98/2	31-113	0-40	0.86	10
2,4-Dimethylphenol	8270(3550)	98/2	10-95	0-25	2.1	10
Dimethylphthalate	8270(3550)	98/2	20-116	0-40	2.1	10
4,6-Dinitro-2-methylphenol	8270(3550)	98/2	43-135	0-93	3.9	50
2,4-Dinitrophenol	8270(3550)	98/2	10-126	0-87	2.8	50

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RL▲ (ug/wipe)
2,4-Dinitrotoluene (MS)	8270(3550)	98/2	26-107	0-30	1.5	10
2,6-Dinitrotoluene	8270(3550)	98/2	38-107	0-40	2.1	10
Di-n-octylphthalate	8270(3550)	98/2	22-135	0-50	0.63	10
Fluoranthene	8270(3550)	98/2	23-121	0-25	0.51	10
Fluorene	8270(3550)	98/2	32-107	0-40	0.60	10
Hexachlorobenzene	8270(3550)	98/2	44-105	0-40	0.44	10
Hexachlorobutadiene	8270(3550)	98/2	10-112	0-40	1.3	10
Hexachlorocyclopentadiene	8270(3550)	98/2	D-132	0-50	4.7	10
Hexachloroethane	8270(3550)	98/2	16-91	0-40	1.7	10
Indeno(1,2,3-cd)pyrene	8270(3550)	98/2	18-157	0-83	0.77	10
Isophorone	8270(3550)	98/2	17-107	0-60	1.9	10
2-Methylnaphthalene	8270(3550)	98/2	19-105	0-50	1.7	10
Naphthalene	8270(3550)	98/2	53-125	0-25	0.47	10
Naphthalene	8270(3550)	98/2	53-125	0-25	0.47	10
2-Nitroaniline	8270(3550)	98/2	22-110	0-50	2.2	50
3-Nitroaniline	8270(3550)	98/2	10-117	0-50	1.9	50
4-Nitroaniline	8270(3550)	98/2	10-136	0-50	2.0	50
Nitrobenzene	8270(3550)	98/2	9-104	0-40	1.6	10
2-Nitrophenol	8270(3550)	98/2	10-101	0-40	2.5	10
4-Nitrophenol (MS)	8270(3550)	98/2	10-117	0-45	2.4	50
N-Nitrosodiphenylamine/ Diphenylamine	8270(3550)	98/2	40-122	0-50	2.6	10
N-Nitroso-di-n-propylamine MS)	8270(3550)	98/2	11-117	0-35	1.4	10
Pentachlorophenol (MS)	8270(3550)	98/2	10-120	0-44	3.2	10
Phenanthrene	8270(3550)	98/2	38-113	0-25	0.92	10
Phenol (MS)	8270(3550)	98/2	17-103	0-25	1.0	10
Pyrene (MS)	8270(3550)	98/2	18-136	0-25	0.89	10
1,2,4-Trichlorobenzene (MS)	8270(3550)	98/2	17-105	0-28	1.4	10
2,4,5-Trichlorophenol	8270(3550)	98/2	39-123	0-27	1.9	10
2,4,6-Trichlorophenol	8270(3550)	98/2	20-116	0-40	1.8	10
Surrogate - Nitrobenzene-d5	8270(3550)	98/2	12-125	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	8270(3550)	98/2	24-118	NA	NA	NA
Surrogate - p-Terphenyl-d14	8270(3550)	98/2	18-153	NA	NA	NA
Surrogate - Phenol-d5	8270(3550)	98/2	10-142	NA	NA	NA
Surrogate - 2-Fluorophenol	8270(3550)	98/2	10-118	NA	NA	NA
Surrogate - 2,4,6-Tribromophenol	8270(3550)	98/2	14-121	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/ wipe)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD)	8280	98/2	69-145	0-40	0.0012	0.0050
Polychlorinated Dibenzo-p-dioxins and Dibenzofurans classes						
tetra-CDD	8280	98/2	69-145	0-40	0.0012	0.0050
tetra-CDF	8280	98/2	59-142	0-40	0.0013	0.0050
penta-CDD	8280	98/2	41-203	0-40	0.0017	0.0050
penta-CDF	8280	98/2	55-146	0-40	0.0016	0.0050
hexa-CDD	8280	98/2	45-174	0-53	0.0011	0.0050
hexa-CDF	8280	98/2	50-154	0-46	0.0015	0.0050
hepta-CDD	8280	98/2	20-170	0-50	0.0018	0.0010
hepta-CDF	8280	98/2	20-170	0-50	0.0013	0.010
octa-CDD	8280	98/2	20-170	0-50	0.0013	0.010
octa-CDF	8280	98/2	20-170	0-50	0.0012	0.010
Internal Standard - 13C12-2,3,7,8-TCDD	8280	98/2	25-150	NA	NA	NA
Internal Standard - 13C12-2,3,7,8-TCDF	8280	98/2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,6,7,8-HxCDD	8280	98/2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,4,6,7,8-HpCDF	8280	98/2	25-150	NA	NA	NA
Internal Standard - 13C12-OCDD	8280	98/2	25-150	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Acenaphthene (MS)	8310(3550)	98/2	11-144	0-35	0.38	1.0
Acenaphthylene	8310(3550)	98/2	10-139	0-40	0.17	1.0
Anthracene	8310(3550)	98/2	10-126	0-40	0.0060	0.20
Benzo(a)anthracene	8310(3550)	98/2	12-135	0-40	0.013	0.20
Benzo(b)fluoranthene	8310(3550)	98/2	10-150	0-40	0.0046	0.20
Benzo(k)fluoranthene	8310(3550)	98/2	10-159	0-40	0.0056	0.20
Benzo(g,h,i)perylene	8310(3550)	98/2	10-120	0-40	0.0035	0.50
Benzo(a)pyrene	8310(3550)	98/2	10-128	0-40	0.062	0.20
Chrysene (MS)	8310(3550)	98/2	10-199	0-40	0.012	0.20
Dibenzo(a,h)anthracene	8310(3550)	98/2	10-110	0-40	0.043	0.50
Fluoranthene	8310(3550)	98/2	56-136	0-28	0.014	0.50
Fluorene (MS)	8310(3550)	98/2	10-142	0-40	0.080	0.50
Indeno(1,2,3-cd)pyrene	8310(3550)	98/2	10-116	0-40	0.019	0.50
1-Methylnaphthalene	8310(3550)	98/2	10-125	0-40	0.15	1.0
2-Methylnaphthalene	8310(3550)	98/2	10-125	0-40	0.16	1.0
Naphthalene (MS)	8310(3550)	98/2	31-159	0-34	0.15	1.0
Phenanthrene	8310(3550)	98/2	10-155	0-40	0.015	0.20
Pyrene (MS)	8310(3550)	98/2	49-156	0-28	0.035	0.50
Surrogate - 4-Terphenyl-d14	8310(3550)	98/2	28-151	NA	NA	NA

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Aluminum (ICP)	6010(3050/3051)	2	75-125	0-20	3.2	20
Antimony (ICP)	6010(3050/3051)	2	75-125	0-20	0.42	2.0
Arsenic (ICP)	6010(3050/3051)	2	75-125	0-20	0.39	1.0
Arsenic (GFAA)	7060(3050/3051)	2	80-120	0-20	0.27	1.0
Barium (ICP)	6010(3050/3051)	2	75-125	0-20	0.42	1.0
Beryllium (ICP)	6010(3050/3051)	2	75-125	0-20	0.11	0.40
Cadmium (ICP)	6010(3050/3051)	2	75-125	0-20	0.056	0.50
Cadmium (GFAA)	7131 (3050/3051)	2	80-120	0-20	0.0090	0.10
Calcium (ICP)	6010(3050/3051)	2	75-125	0-20	2.4	50
Chromium (ICP)	6010(3050/3051)	2	75-125	0-20	0.44	1.0
Cobalt (ICP)	6010 (3050/3051)	2	75-125	0-20	0.13	1.0
Copper (ICP)	6010 (3050/3051)	2	75-125	0-20	0.22	2.0
Iron (ICP)	6010 (3050/3051)	2	75-125	0-20	0.88	5.0
Lead (ICP)	6010 (3050/3051)	2	75-125	0-20	0.20	050
Lead (GFAA)	7421(3050/3051)	2	80-120	0-20	0.14	0.50
Magnesium (ICP)	6010 (3050/3051)	2	75-125	0-20	1.7	50
Manganese (ICP)	6010(3050/3051)	2	75-125	0-20	0.18	1.0
Mercury (CVAA)	7471 (3050/3051)	2	80-120	0-20	0.0020	0.020
Molybdenum (ICP)	6010(3050/3051)	2	75-125	0-20	0.23	1.0
Nickel (ICP)	6010(3050/3051)	2	75-125	0-20	1.1	4.0
Potassium (ICP)	6010(3050/3051)	2	75-125	0-20	35	100
Selenium (ICP)	6010 (3050/3051)	2	75-125	0-20	0.46	1.0
Selenium (GFAA)	7740(3050/3051)	2	80-120	0-20	0.13	1.0
Silver (ICP)	6010 (3050/3051)	2	75-125	0-20	0.14	1.0
Silver (GFAA)	7761 (3050/3051)	2	80-120	0-20	0.016	0.10
Sodium (ICP)	6010(3050/3051)	2	75-125	0-20	8.3	50
Strontium(ICP)	6010(3050/3051)	2	75-125	0-30	0.84	1.0
Thallium (ICP)	6010 (3050/3051)	2	75-125	0-20	0.46	1.0
Thallium (GFAA)	7841 (3050/3051)	2	80-120	0-20	0.14	1.0
Tin (ICP)	6010(3050/3051)	2	75-125	0-20	1.3	5.0
Titanium	6010(3050/3051)	2	75-125	0-20	0.25	1.0
Vanadium (ICP)	6010(3050/3051)	2	75-125	0-20	0.23	1.0
Zinc (ICP)	6010(3050/3051)	2	75-125	0-20	0.45	2.0

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTE AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Cyanide, reactive	7.3.3.2	2	NA	0-50	NA	10mg HCN Kg Waste
Cyanide, total	9012(9013)	2	75-125	0-30	0.036	1.0
	9012(9010)	2	75-125	0-30	0.065	1.0
pH	9041	2	63-158	0-40	NA	NA

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Aldrin (MS)	8081(3580)	2	10-168	0-38	0.63	50
alpha-BHC	8081(3580)	2	37-134	0-40	2.6	50
beta-BHC	8081(3580)	2	17-147	0-40	4.8	50
gamma-BHC (Lindane) (MS)	8081(3580)	2	10-173	0-37	2.0	50
delta-BHC	8081(3580)	2	19-140	0-47	5.4	50
technical Chlordane	8081(3580)	2	45-119	0-40	51	500
alpha Chlordane	8081(3580)	2	45-140	0-40	3.6	50
gamma Chlordane	8081(3580)	2	45-140	0-40	2.3	50
4,4'-DDD	8081(3580)	2	31-140	0-50	6.6	100
4,4'-DDE	8081(3580)	2	30-145	0-25	3.9	100
4,4'-DDT (MS)	8081(3580)	2	10-181	0-26	15	100
Dieldrin (MS)	8081(3580)	2	10-176	0-30	5.7	100
Endosulfan I	8081(3580)	2	45-153	0-40	3.9	50
Endosulfan II	8081(3580)	2	10-202	0-65	6.6	100
Endosulfan sulfate	8081(3580)	2	26-144	0-50	27	100
Endrin (MS)	8081(3580)	2	10-180	0-32	3.0	100
Endrin aldehyde	8081(3580)	2	10-150	0-86	6.6	100
Endrin ketone	8081(3580)	2	40-100	0-31	13	100
Heptachlor (MS)	8081(3580)	2	10-162	0-38	3.6	50
Heptachlor epoxide	8081(3580)	2	37-142	0-40	3.0	50
Methoxychlor	8081(3580)	2	34-166	0-40	39	500
Toxaphene	8081(3580)	2	41-126	0-50	540	5000

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
PCB-1016	8082(3580)	2	52-152	0-44	140	1000
	EPA-600/4-81-045	61	50-130	0-50	1200	5000
PCB 1221	8082(3580)	2	15-178	0-30	690	1000
	EPA-600/4-81-045	61	50-130	0-50	1200	5000
PCB 1232	8082(3580)	2	10-215	0-30	120	1000
	EPA-600/4-81-045	61	50-130	0-50	1200	5000
PCB-1242	8082(3580)	2	39-150	0-30	180	1000
	EPA-600/4-81-045	61	50-130	0-50	1200	5000
PCB-1248	8082(3580)	2	38-158	0-30	200	1000
	EPA-600/4-81-045	61	50-130	0-50	1200	5000
PCB-1254	8082(3580)	2	66-122	0-30	81	1000
	EPA-600/4-81-045	61	50-130	0-50	1200	5000
PCB-1260	8082(3580)	2	58-150	0-30	170	1000
	EPA-600/4-81-045	61	50-130	0-50	1200	5000
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081(3580)/8082(3580)	2	10-114	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	8081(3580)/8082(3580)	2	27-128	NA	NA	NA

Waste dilution extraction : 1g of sample to a final volume of 10mL with appropriate solvent.

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Polychlorinated biphenyl classes						
Monochlorobiphenyls	680(3580)	93/2	30-130	0-50	20	300
Dichlorobiphenyls	680(3580)	93/2	30-130	0-50	23	300
Trichlorobiphenyls	680(3580)	93/2	30-130	0-50	20	300
Tetrachlorobiphenyls	680(3580)	93/2	40-140	0-50	39	600
Pentachlorobiphenyls	680(3580)	93/2	40-140	0-50	25	600
Hexachlorobiphenyls	680(3580)	93/2	40-140	0-50	27	600
Heptachlorobiphenyls	680(3580)	93/2	40-140	0-50	48	900
Octachlorobiphenyls	680(3580)	93/2	40-140	0-50	29	900
Nonochlorobiphenyls	680(3580)	93/2	30-130	0-50	57	1500
Decachlorobiphenyl	680(3580)	93/2	30-130	0-50	57	1500
Surrogate - $^{13}C_{12}$-Decachlorobiphenyl	680(3580)	93/2	30-130	NA	NA	NA

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acetone	8260(5035ext)	2	14-189	00-40	1200	25000
Benzene (MS)	8260(5035ext)	2	64-144	0-25	210	2500
Bromodichloromethane	8260(5035ext)	2	71-140	0-40	170	2500
Bromoform	8260(5035ext)	2	59-143	0-40	430	2500
Bromomethane (Methyl bromide)	8260(5035ext)	2	35-181	0-65	1000	5000
2-Butanone (MEK)	8260(5035ext)	2	54-166	0-40	1400	12500
Carbon disulfide	8260(5035ext)	2	10-160	0-65	150	2500
Carbon tetrachloride	8260(5035ext)	2	65-129	0-40	240	2500
Chlorobenzene (MS)	8260(5035ext)	2	56-152	0-25	290	2500
Chloroethane	8260(5035ext)	2	65-129	0-40	650	5000
Chloroform	8260(5035ext)	2	62-117	0-40	130	2500
Chloromethane	8260(5035ext)	2	10-186	0-65	600	5000
Dibromochloromethane	8260(5035ext)	2	73-127	0-40	140	2500
1,1-Dichloroethane	8260(5035ext)	2	64-112	0-40	140	2500
1,2-Dichloroethane	8260(5035ext)	2	66-128	0-40	500	2500
cis-1,2-Dichloroethene	8260(5035ext)	2	64-123	0-40	160	2500
trans-1,2-Dichloroethene	8260(5035ext)	2	60-161	0-25	290	2500
1,1-Dichloroethene (MS)	8260(5035ext)	2	44-157	0-25	390	2500
1,2-Dichloropropane	8260(5035ext)	2	47-146	0-65	500	2500
cis-1,3-Dichloropropene	8260(5035ext)	2	51-109	0-65	250	2500
trans-1,3-Dichloropropene	8260(5035ext)	2	57-143	0-65	330	2500
Ethylbenzene	8260(5035ext)	2	60-131	0-40	280	2500
2-Hexanone	8260(5035ext)	2	47-145	0-40	1900	12500
Methylene chloride	8260(5035ext)	2	61-149	0-65	330	2500
4-Methyl-2-pentanone (MIBK)	8260(5035ext)	2	64-167	0-49	1600	12500
Styrene	8260(5035ext)	2	56-121	0-40	2220	2500
1,1,2,2-Tetrachloroethane	8260(5035ext)	2	36-157	0-40	400	2500
Tetrachloroethene	8260(5035ext)	2	61-142	0-40	220	2500
Toluene (MS)	8260(5035ext)	2	67-142	0-25	240	2500
1,1,1-Trichloroethane	8260(5035ext)	2	11-148	0-40	490	2500
1,1,2-Trichloroethane	8260(5035ext)	2	66-123	0-40	330	2500
Trichloroethene (MS)	8260(5035ext)	2	41-134	0-25	360	2500
Vinyl chloride	8260(5035ext)	2	18-169	0-65	490	5000
Xylenes (total)	8260(5035ext)	2	50-150	0-40	750	2500
o-Xylene	8260(5035ext)	2	22-154	0-40	250	2500
m+p-Xylene	8260(5035ext)	2	62-123	0-40	500	2500
Surrogate - Toluene-d8	8260(5035ext)	2	58-148	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5035ext)	2	70-136	NA	NA	NA
Surrogate -Dibromofluoromethane	8260(5035ext)	2	66-148	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	8260(5035ext)	2	70-130	NA	NA	NA

ext= 1g of waste to 10mL methanol; analyze 0.10mL of extract (equivalent to 0.010g of sample)

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acenaphthene (MS)	8270(3580)	2	28-102	0-25	3900	100000
Acenaphthylene	8270(3580)	2	54-140	0-25	3900	100000
Anthracene	8270(3580)	2	48-130	0-30	3600	100000
Benzo(a)anthracene	8270(3580)	2	42-143	0-25	2900	100000
Benzoic acid	8270(3580)	2	10-150	0-50	6300	500000
Benzo(b)fluoranthene	8270(3580)	2	49-123	0-25	5100	100000
Benzo(k)fluoranthene	8270(3580)	2	24-137	0-38	3900	100000
Benzo(g,h,i)perylene	8270(3580)	2	10-219	0-50	5700	100000
Benzo(a)pyrene	8270(3580)	2	44-141	0-29	3300	100000
Bis(2-chloroethoxy) methane	8270(3580)	2	33-184	0-50	9000	100000
Bis(2-chloroethyl) ether	8270(3580)	2	12-158	0-50	2900	100000
Bis(2-chloroisopropyl) ether	8270(3580)	2	36-166	0-50	3000	100000
Bis(2-ethylhexyl) phthalate	8270(3580)	2	10-158	0-40	14000	100000
4-Bromophenyl phenyl ether	8270(3580)	2	53-127	0-40	3300	100000
Butyl benzyl phthalate	8270(3580)	2	10-152	0-40	9000	100000
Carbazole	8270(3580)	2	10-150	0-50	4200	100000
4-Chloroaniline	8270(3580)	2	10-150	0-50	5100	200000
4-Chloro-3-methylphenol (MS) (p-Chloro-m-cresol)	8270(3580)	2	31-123	0-25	3000	100000
2-Chloronaphthalene	8270(3580)	2	60-118	0-40	3300	100000
2-Chlorophenol (MS)	8270(3580)	2	29-108	0-25	3300	100000
4-Chlorophenylphenyl ether	8270(3580)	2	25-158	0-33	3000	100000
Chrysene	8270(3580)	2	40-148	0-27	3600	100000
2-Methyl phenol (o-Cresol)	8270(3580)	2	10-150	0-50	3900	100000
3-Methyl phenol (m-Cresol)	8270(3580)	2	10-150	0-50	18000	100000
4-Methyl phenol (p-Cresol)	8270(3580)	2	10-150	0-50	18000	100000
Dibenz(a,h)anthracene	8270(3580)	2	40-147	0-28	4500	100000
Dibenzofuran	8270(3580)	2	10-150	0-50	3600	100000
Di-n-butylphthalate	8270(3580)	2	10-118	0-50	4500	100000
1,2-Dichlorobenzene	8270(3580)	2	32-129	0-40	3300	100000
1,3-Dichlorobenzene	8270(3580)	2	10-172	0-42	2600	100000
1,4-Dichlorobenzene (MS)	8270(3580)	2	20-114	0-40	3300	100000

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
3,3'-Dichlorobenzidine	8270(3580)	2	10-262	0-100	4800	200000
2,4-Dichlorophenol	8270(3580)	2	39-135	0-40	3000	100000
Diethylphthalate	8270(3580)	2	10-114	0-40	4500	100000
2,4-Dimethylphenol	8270(3580)	2	15-151	0-25	3000	100000
Dimethylphthalate	8270(3580)	2	10-112	0-40	3300	100000
4,6-Dinitro-2-methylphenol	8270(3580)	2	10-181	0-93	8700	500000
2,4-Dinitrophenol	8270(3580)	2	10-167	0-87	6600	500000
2,4-Dinitrotoluene (MS)	8270(3580)	2	25-128	0-30	3900	100000
2,6-Dinitrotoluene	8270(3580)	2	50-158	0-40	4800	100000
Diphenylamine/ N-nitrosodiphenylamine	8270(3580)	2	10-150	0-50	29000	100000
Fluoranthene	8270(3580)	2	54-135	0-21	3300	100000
Fluorene	8270(3580)	2	59-121	0-40	3600	100000
Hexachlorobenzene	8270(3580)	2	10-152	0-40	3000	100000
Hexachlorobutadiene	8270(3580)	2	24-116	0-40	3300	100000
Hexachlorocyclopentadiene	8270(3580)	2	10-150	0-50	5400	100000
Hexachloroethane	8270(3580)	2	40-113	0-40	3900	100000
Indeno(1,2,3-cd)pyrene	8270(3580)	2	18-157	0-83	6000	100000
Isophorone	8270(3580)	2	21-196	0-60	3600	100000
2-Methylnaphthalene	8270(3580)	2	10-150	0-50	2800	100000
Naphthalene	8270(3580)	2	53-125	0-25	3300	100000

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (prep)	REF	ACCURACY* (%Rec)	PRECISION * (%RPD)	MDL** (ug/kg)	RLA (ug/kg
2-Nitroaniline	8270(3580)	2	10-150	0-50	4200	500000
3-Nitroaniline	8270(3580)	2	10-150	0-50	3300	500000
4-Nitroaniline	8270(3580)	2	10-150	0-50	3900	500000
Nitrobenzene	8270(3580)	2	35-180	0-40	3900	100000
2-Nitrophenol	8270(3580)	2	29-182	0-40	2400	100000
4-Nitrophenol (MS)	8270(3580)	2	10-125	0-45	9000	500000
N-Nitroso-di-n-propylamine (MS)	8270(3580)	2	27-140	0-35	4200	100000
Pentachlorophenol (MS)	8270(3580)	2	10-111	0-44	8400	500000
Phenanthrene	8270(3580)	2	56-129	0-25	3600	100000
Phenol (MS)	8270(3580)	2	26-108	0-25	5700	100000
Pyrene (MS)	8270(3580)	2	24-155	0-25	3300	100000
1,2,4-Trichlorobenzene (MS)	8270(3580)	2	23-124	0-28	3300	100000
2,4,5-Trichlorophenol	8270(3580)	2	39-123	0-27	3900	100000
2,4,6-Trichlorophenol	8270(3580)	2	37-144	0-40	2800	100000
Surrogate - Nitrobenzene-d5	8270(3580)	2	21-98	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	8270(3580)	2	29-99	NA	NA	NA
Surrogate - p-Terphenyl-d14	8270(3580)	2	22-136	NA	NA	NA
Surrogate - Phenol-d5	8270(3580)	2	28-105	NA	NA	NA
Surrogate - 2-Fluorophenol	8270(3580)	2	18-107	NA	NA	NA
Surrogate - 2,4,6 - Tribromophenol	8270(3580)	2	12-122	NA	NA	NA

Waste dilution extraction : 1g of sample ot a final volume of 10mL with appropriate solvent.

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD)	8280	2	69-145	0-40	1.3	50
Polychlorinated Dibenzo-p-dioxins and Dibenzofurans classes						
tetra-CDD	8280	2	69-145	0-40	1.2	50
tetra-CDF	8280	2	59-142	0-40	0.40	50
penta-CDD	8280	2	41-203	0-40	0.89	50
penta-CDF	8280	2	55-146	0-40	0.68	50
hexa-CDD	8280	2	45-174	0-53	1.3	50
hexa-CDF	8280	2	50-154	0-46	0.51	50
hepta-CDD	8280	2	20-170	0-50	1.3	100
hepta-CDF	8280	2	20-170	0-50	0.63	100
octa-CDD	8280	2	20-170	0-50	1.4	100
octa-CDF	8280	2	20-170	0-50	1.3	100
Internal Standard - 13C12-2,3,7,8-TCDD	8280	2	25-150	NA	NA	NA
Internal Standard - 13C12-2,3,7,8-TCDF	8280	2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,6,7,8-HxCDD	8280	2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,4,6,7,8-HpCDF	8280	2	25-150	NA	NA	NA
Internal Standard - 13C12-OCDD	8280	2	25-150	NA	NA	NA

TABLE 5.8 FIELD ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Chlorine, residual	4500-Cl^-F	4	NA	0-40	NA	0.05
	330.5/4500-Cl-G (Hach8021)	3/4	NA	0-40	NA	0.05
Hydrogen ion (pH)	150.1/9040	3/2	85-115	0-15	NA	NA
Oxygen (dissolved)	360.1	3	NA	0-30	NA	0.20
Salinity	2520B	4	NA	NA	NA	100
Specific conductance	120.1/9050	3/2	90-110	0-10	0.26 µS/cm	1.0 µS/cm
Temperature	170.1	3	NA	0-10	NA	NA
Turbidity	180.1/2130B	3/4	60-140	0-30	0.008 NTU	0.10 NTU
Water level	EPA	12	NA	0-5	NA	0.01 ft
Sulfite	4500-SO_3^{2-}B	4	75-125	0-30	NA	1.0
	377.1	3	75-125	0-30	NA	1.0

REFERENCES AND NOTES FOR TABLES 5.1, 5.2, 5.3, 5.4, 5.5, 5.6, 5.7, 5.8

* Accuracy data are presented as recoveries for spikes or surrogates. For routine analysis of organics, percent recoveries are evaluated only on the subset spike compound lists specified by the methods. An (MS) following the parameter name designates the routine matrix and laboratory control spike compounds. Precision data are presented as relative percent difference (% RPD) and are advisory; i.e., not used for laboratory control. Since reportable levels (above detection limit) for most of the organic parameters may not be detected in all environmental samples, precision is usually based on duplicate spike data and evaluated according to method requirements.

Accuracy and precision control limits are primarily derived from in-house laboratory data. Some accuracy and precision limits have been rounded to the nearest "5". In some cases, method limits may be used in lieu of in-house limits because in-house limits are broader than the method limits or are too broad to be usable. These cases include all metals methods, certain of the 500-series methods, method 8015 DAI, and all 600 series methods. In-house accuracy and precision limits in Tables 5.1 - 5.8 have been developed from spike concentrations between 0.5 times to 10 times the routine reporting (quantitation) limits.

** Method Detection Limit (MDL)

A Routine Reporting Limit (also referred to as quntitation limt or pratical quantitation limit(PQL).)

NA Not Applicable

SIM (Selected ion monitoring.) Savannah Laboratories has verified that the analytes listed can be determined using this procedure.

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6.0 SAMPLING PROCEDURES

When Savannah Laboratories is contracted to provide sampling services, a field crew is assigned to each project. Each crew is composed of experienced field sampling technicians and a highly qualified field sampling manager who is trained in EPA protocols for groundwater and other environmental sampling. On numerous past projects, these manager have had their field sampling techniques audited by Florida DEP, Georgia EPD, Alabama ADEM, South Carolina DHEC, and EPA Region IV QA or field personnel. The field sampling crews at Savannah Laboratories are responsible for collection, handling, field screening, documentation, and packaging and shipment of samples to the lab in accordance with client requests.

The crew adheres to the sampling protocol defined by the appropriate regulatory agency or the requirements defined in the project sampling plan (QAPP). In some cases, sampling procedures may be modified to comply with client" project requirements.

6.1 Sampling Capabilities

Savannah Laboratories has the capability for sampling groundwater, surface water, wastewater, soils, sediments/sludges, drinking water, and tissues for the following analyte classes:

Analyte Class	Sample Source
Volatile Organics (VOAs)	Drinking water, groundwater, surface water, wastewater, sols, sediments, fish, shellfish, plant and animal tissues, liquid hazardous wastes, sludges, solid and hazardous wastes, and domestic waste sludges.
Extractable Organics	Drinking water, groundwater, surface water, wastewater, soils, sediments, fish, shellfish, plant and animal tissues, liquid hazardous wastes, sludges, solid and hazardous wastes, and domestic waste sludges.
Metals	Drinking water, groundwater, surface water, wastewater, soils, sediments, fish, shellfish, plant and animal tissues, liquid hazardous wastes, sludges, solid and hazardous wastes, and domestic waste sludges.
Radionuclides	Drinking water, groundwater, surface water, wastewater, soils, and sediments
Microbiology	Drinking water, groundwater, surface water, wastewater, soils, sediments, and tissues
Cyanide/Sulfide	Drinking water, groundwater, surface water, wastewater, soils, sediments, liquid hazardous wastes, sludges, solid and hazardous waste, and domestic waste sludges.
Inorganic Anions[1]	Drinking water, groundwater, surface water, wastewater, soils, sediments, liquid hazardous wastes, sludges, solid and hazardous waste, and domestic waste sludges.
Organics: TOC, COD, BOD, Total Recoverable Petroleum Hydrocarbons, Oil & Grease, Phenolics, MBAS	Drinking water, groundwater, surface water, wastewater, soils, sediments, liquid hazardous wastes, sludges, solid and hazardous waste, and domestic waste sludges.
Physical Properties: Color, Specific Conductance, Hardness, Odor, pH, Residues, Temperature. Turbidity	Drinking water, groundwater, surface water, wastewater, soils, sediments, liquid hazardous wastes, sludges, solid and hazardous waste, and domestic waste sludges.

[1]Nitrogen, Phosphorus Series, Bromide, Chloride, Chlorine, Iodide, Sulfate, Acidity, Alkalinity, DO, IC Parameters

6.2 Sampling Equipment

The following is a list of equipment and the sample preservation reagents employed by Savannah Laboratories' field sampling crews.

Routinely Used Equipment	Use
Ice chests, Styrofoam or insulated plastic	Sample container and sample transport
Sampling vehicles	Sample container and sample transport
Field thermometer	Field measurement of temperature
Field pH meter	Field measurement of pH
Field conductivity meter	Field measurement of conductivity
Electronic water level indicator	Well volume calculation
Stainless steel tape measure	Well volume calculation
Nylon, monofilament, or polypropylene line	Well volume calculation
Sheet plastic	Contamination control
Aluminum foil	Contamination control
Plastic or metal buckets	Collection of purge water or cleaning wastes
Cleaning brushes	Equipment decontamination
Liquinox detergent in original container	Equipment decontamination
Analyte free water contained in contaminant-free glass or plastic bottles	Equipment decontamination
Isopropyl alcohol (nanograde) contained in contaminant-free glass bottles or Teflon squeeze bottles	Equipment decontamination
10% Nitric acid (metals grade) contained in contaminant-free glass bottles	Equipment decontamination (except for stainless steel equipment)
Glass or plastic bottles and dispensers	Equipment decontamination
Glass or plastic jugs	Transport of cleaning wastes
Field carrier (covered, divided tray or box)	Transport of preservation reagents
Narrow range pH paper	Field check of sample preservation
Disposable pipettes glass (organic) and plastic (inorganic)	Addition of preservation reagents
Standard buffer solutions (pH 4,7, and 10)	Calibration of field pH meter
Standard KCl solution(100, 500, 1000, 1413, 12880 micro ohms/cm)	Calibration check of field conductivity and salinity meter
Disposable unpowdered latex gloves	Contamination control
Ice	Sample preservation
Sealing Tape	Sealing sample containers (except VOA vials)
Shipping labels and forms	Shipping samples
Sample container labels	Labeling samples
Bubble pack	Packing samples
Clothing and goggles	Sampling safety
Notebooks	Documentation
Waterproof pens, markers	Documentation, labeling
Custody seals	Monitor for tampering
Custody forms	Document custody
Camera	Document site
Calculator	Calculations
Site maps	Determine locations
SOPs	Reference procedures
MSDS Sheet on all chemicals	Safety emergency
Turbidity meter with 0.75 , 10, and 100 NTU standards	Field measurement of turbidity
Chlorine test kit with pocket colorimeter	Field determination of chlorine
Sulfite test kit with calibration burets	Field determination of sulfite
Paper towels	General use

Preservation Reagents	Grade
HCl,1:1	Metals grade, pre-assayed
HNO_3, 1:1	Metals grade, pre-assayed
H_2SO_4, 1:1	Metals grade, pre-assayed
NaOH	ACS reagent grade
$Na_2S_2O_3$, 10%, 0.008%	ACS reagent grade
$Zn(C_2H_3O_2)_2$, 2N	ACS reagent grade
Ascorbic acid, neat; 0.06%	ACS reagent grade
Chloroacetic acid	ACS reagent grade

6.3 Routine Sample Containers

The following tables list the parameter, the routine container, the chemical preservative required to maintain the integrity of the sample, the hold time (preparation and analytical), and the minimum volume or weight of sample required to complete the analysis. The preparation of sampling kits is described in SL SOP CU15: *Preparation of Sampling Containers*. A table of contents for these tables is given below.

The Routine Containers used for sampling and analysis are certified by SL to ensure that the parameters tested are below the reporting limits published in the current SL CQAP. The procedures for certifying containers are given in SL SOP CU35: *Procedures for Contaminant-free Containers*.

The chemical preservative is the solution added to the sampling containers or supplied as separate solutions or neat materials that preserve the integrity of the sample. Chemical preservatives are generally analyte specific. Generally, parameters requiring the same type of container and preservation can be analyzed from the same container. In addition to chemical preservation, the samples must be iced at the time of collection and maintained in the laboratory at the required temperature, usually 0-4C (control limits of less than 6C with no frozen samples).

The hold time is the maximum time from collection that the sample can be held prior to preparation or analysis. Some parameters, for example, semivolatile organics, have separate hold times for preparation of the sample and analysis of the extract.

The minimum volume or weight represents the volume of sample required to analyze for the parameter *one time* without sample matrix spikes to achieve the routine reporting levels. If smaller volumes or weights of sample are supplied that those listed in the table, the reporting limit will be elevated to reflect the sample size. If matrix spikes are required, the minimum volume or weight of sample required is three times the volume or weight listed in the table.

The routine reporting limit for some analyses can be attained when a less than optimum sample volume or weight is supplied. For some analyses (e.g., organic extractions), the final volume of the extract or digest can be adjusted to met the routine reporting limits. If a client requires a normal reporting limit from a reduced sample amount, the client must inform their project manager, who will alert the lab prior to sample analysis because the amount of surrogate and/or spiking solution must be adjusted prior to sample preparation.

The minimum volume or weight required for TCLP is determined by the procedure. The lab must have these minimum amounts of sample in order to meet the Regulatory Threshold Limits for hazardous wastes. If less than the required weight or volume is supplied, the data may be flagged or the results for the TCLP invalidated by the regulatory agency.

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ANALYSES/CONTAINERS FOR LIQUIDS -General Chemistry

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	Hold Time	MINIMUM VOLUME REQUIRED
Acidity	120-ml P	none	14 days	50mL
Alkalinity	120-ml P	none	14 days	50mL
Bromide (IC)	120-ml P	none	28 days	10mL
Chloride (autoanalyzer)	120-ml P	none	28 days	10mL
Chloride (IC)	120-ml P	none	28 days	10mL
Color	120-ml P	none	2 days	50mL
Fluoride (electrode)	120-ml P	none	28 days	25mL
Fluoride (IC)	120-ml P	none	28 days	10mL
Hydrogen ion	120-ml P	none	analyze ASAP	25mL
Nitrate (autoanalyzer)	120-ml P	none	2 days	10mL
Nitrate (IC)	120-ml P	none	2 days	10mL
Nitrite (colorimetric)	120-ml P	none	2 days	10mL
Nitrite (IC)	120-ml P	none	2 days	10mL
Orthophosphate	120-ml P	none	2 days	25mL
Specific Conductance	120-ml P	none	28 days	25mL
Sulfate (turbidimetric)	120-ml P	none	28 days	25mL
Sulfate (IC)	120-ml P	none	28 days	10mL
Sulfite	120-ml P	none	analyze ASAP	50mL
Temperature	120-ml P	none	analyze ASAP	10mL
Turbidity	120-ml P	none	2 days	25mL
Ammonia	120-ml P	0.5mL 1:1 sulfuric acid	28 days	10mL
Ammonia (if distillation required)	120-ml P	0.5mL 1:1 sulfuric acid	28 days	25mL
COD	120-ml P	0.5mL 1:1 sulfuric acid	28 days	25mL
Nitrate – Nitrite	120-ml P	0.5mL 1:1 sulfuric acid	28 days	10mL
Phosphorus, total	120-ml P	0.5mL 1:1 sulfuric acid	28 days	25mL
Hexavalent chromium	250-ml P	none	1 day	10mL
Cyanide, reactive	250-mL P	none	14 days	10ml./10g

(1) Containers should be iced at time of collection in addition to the chemical preservation

ANALYSES/CONTAINERS FOR LIQUIDS-General Chemistry

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Kjeldahl (TKN) and organic nitrogen	250-ml P	1mL 1:1 sulfuric acid		25mL
Hardness (EDTA titration)	250-ml P	1mL 1:1 sulfuric acid		100mL
Ferrous Iron (colorimetric)	250-ml P	1 mL 1:1 hydrochloric acid	analyze ASAP	25mL
BOD	500-ml P	none	2 days	100mL
Residue, total	500-ml P	none	7 days	100mL
Residue, filterable (TDS)	500-ml P	none	7 days	100mL
Residue, non-filterable (TSS)	500-ml P	none	7 days	100mL
Residue, settable	500-ml P	none	2 days	100mL
Residue, volatile (VSS)	500-ml P	none	7 days	100mL
Surfactants	500-ml P	none	2 days	100mL
Sulfide, reactive	500-ml P	none		10mL (10g)
Sulfide	500-ml P	2mL 2N zinc acetate	7 days	250mL
Cyanide, total and amenable to chlorination	250-mL P 500-mL P	4-5 pellets sodium hydroxide 8-10 pellets sodium hydroxide	14 days	50mL
Cyanide CLP ILMO 2.1/3.0/4.0	250-mL P	4-5 pellets sodium hydroxide	12 days (2)	50mL
Organic Carbon (TOC) (Shimadzu analyzer-Mobile and Savannah)	125-ml amb G	1mL 1:1 hydrochloric acid	28 days	10mL
Organic Carbon (TOC) (Dohrmann analyzer-Tallahassee)	125-ml amb G	1mL 1:1 sulfuric acid	28 days	10mL
Chlorine, total residual	250-ml amb G	none	analyze ASAP	25mL

(1) Containers should be iced at time of collection in addition to the chemical preservation
(2) The hold time for CLP methods is measured from the date of receipt into the laboratory.

ANALYSES/CONTAINERS FOR LIQUIDS-General Chemistry

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Oxygen, dissolved (Winkler)	BOD Bottle G	HACH DO reagent powder pillows	analyze ASAP	full BOD bottle
Oxygen, dissolved (electrode)	BOD Bottle G	none	analyze ASAP	full BOD bottle
Absorbable organic halides (AOX)	500-ml amb G	2mL 1:1 nitric acid	28 days	100mL
Total organic halogens (TOX)	500-ml amb G	2mL 1:1 sulfuric acid	28 days	100mL
Phenol, total recoverable (direct)	125-mL amber G	1mL 1:1 sulfuric acid	28 days	50mL
Phenols, total recoverable (chloroform extraction)	500-ml amb G	2mL 1:1 sulfuric acid	28 days	300mL

ANALYSES/CONTAINERS FOR LIQUIDS-General Chemistry/Extraction La

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(2)	HOLD TIME	MINIMUM VOLUME REQUIRED
Oil and Grease (gravimetric) (418.2)	500-mL or 1-L Glass (1)	2mL 1:1 sulfuric acid	28 days	1-L
Hexane Extractable Materials (HEM) (1664)	1-L Glass (1)	2mL 1:1 sulfuric acid	28 days	1-L
Total petroleum hydrocarbons and oil and grease by IR (418.1)	125-ml amb G (1)	2mL 1:1 sulfuric acid	28 days	125mL
Surfactants	500-mL P	none	48 hours	100mL

(1) The entire contents of each container must be used for analysis.
(2) Containers should be iced at time of collection in addition to the chemical preservation

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS -General Chemistry

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	Hold Time	MINIMUM WEIGHT REQUIRED
Ammonia	250-mL P	none	28 days	10g
BOD	500-ml P	none	2 days	100g
Bromide (IC)	250-mL P	none	28 days	10g
COD	250-mL P	none	28 days	10g
Chloride (autoanalyzer)	250-mL P	none	28 days	10g
Chloride (IC)	250-mL P	none	28 days	10g
Cyanide, total and amenable to chlorination	250-mL P	none	14 days	1g
Cyanide (CLP ILMO 2.1/3.0/4.0)	250-mL P	none	12 days (2)	1g
Cyanide, reactive	250-mL P	none	14 days	10mL/10g
Fluoride (electrode)	250-mL P	none	28 days	10g
Fluoride (IC)	250-mL P	none	28 days	10g
Hydrogen ion	250-mL P	none	Analyze ASAP	10g
Kjeldahl (TKN) and organic nitrogen	250-mL P	none	28 days	10g
Nitrate (autoanalyzer)	250-mL P	none	28 days	10g
Nitrate (IC)	250-mL P	none	28 days	10g
Nitrite (colorimetric)	250-mL P	none	28 days	10g
Nitrite (IC)	250-mL P	none	28 days	10g
Organic Carbon	250-mL P	none	28 days	10g
Orthophosphate	250-mL P	none	28 days	10g
Phosphorus, total	250-mL P	none	28 days	10g
Specific Conductance	250-mL P	none	28 days	10g
Sulfate (turbidimetric)	250-mL P	none	28 days	10g
Sulfate (IC)	250-mL P	none	28 days	10g
Residue, total	250-mL P	none	28 days	10g
Residue, volatile (VSS)	250-mL P	none	28 days	10g
Sulfide	250-mL P	none	28 days	10g
Sulfide, reactive	250-mL P	none	28 days	10g
Surfactants	250-mL P	none	28 days	10g

(1) Containers should be iced at time of collection.
(2) The hold time for CLP methods is measured from the date of receipt into the laboratory.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-General Chemistry

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM WEIGHT REQUIRED
Extractable organic halogens (EOX)	125-mL amber G	none	28 days	1g
Phenol, total recoverable (direct)	125-mL amber G	none	28 days	5g
Phenols, total recoverable (chloroform extraction)	125-mL amber g	none	28 days	5g

(1) Containers should be iced at time of collection.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-General Chemistry/Extraction Lab

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM WEIGHT REQUIRED
Oil and Grease (gravimetric)	250-mL or 500-mL amb G	None	28 days	30g
Total petroleum hydrocarbons and oil and grease by IR	250-mL or 500-mL amb G	None	28 days	30g

(1) Containers should be iced at time of collection.

CONTAINERS FOR LIQUIDS AND SOILS-General Chemistry

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Nitrocellulose (water)	1-L amb G	None	7 days/40 days(2)	1-L
Nitrocellulose (soils)	500-mL amb G	None	40days/1 day(2)	100g

(1) Containers should be iced at time of collection in addition to the chemical preservation

(2) The first number is the hold time until the extraction; the second time is the hold time for the extract; i.e., the extraction must take place within 7 days of collection and the extract must be analyzed within 40 days of the date of extraction.

ANALYSES/CONTAINERS FOR LIQUIDS-Metals

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME	MINIMUM VOLUME REQUIRED
Mercury	250-ml P or 500-mL P	1mL 1:1 nitric acid 2mL 1:1 nitric acid	28 days(1)	50mL
Metals (except Hexavalent Chromium and Mercury)	250 ml P or 500-mL P	1mL 1:1 nitric acid 2mL 1:1 nitric acid	6 months	50mL
Mercury CLP ILMO 2.1/3.0/4.0	250-ml P or 500-mL P	1mL 1:1 nitric acid 2mL 1:1 nitric acid	26 days(1,2)	50mL
Metals CLP ILMO 2.1/3.0/4.0	250 ml P or 500-mL P	1mL 1:1 nitric acid 2mL 1:1 nitric acid	6 months(2)	50mL
Hexavalent chromium	250-ml P	none	1 day	10mL
Ferrous Iron (colorimetric)	250-ml P	1 mL 1:1 hydrochloric acid	analyze ASAP	25mL

(1) Containers should be iced at time of collection in addition to the chemical preservation.
(2) The hold time for CLP methods is measured from the date of receipt into the laboratory.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-Metals

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME	MINIMUM WEIGHT REQUIRED
Mercury	250-ml P or 500-mL P	none(1)	28 days	5g
Metals (except Chromium VI and Mercury)	250 ml P or 500-mL P	none(1)	6 months	5g
Mercury CLP ILMO 2.1/3.0/4.0	250-ml P or 500-mL P	none(1)	26 days (2)	5g
Metals CLP ILMO 2.1/3.0/4.0	250 ml P or 500-mL P	none(1)	6 months	5g
Hexavalent chromium	250-ml P or 500-mL P	None(1)	30 day/ 7 days (3)s	10mL

(1) Containers should be iced at time of collection.
(2) The hold time for CLP methods is measured from the date of receipt into the laboratory.
(3) The digestion must be completed within 30 days and the alkaline digestate must be analyzed within seven days of the digestion.

ANALYSES/CONTAINERS FOR LIQUIDS-VOC by GC

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME (3)	MINIMUM VOLUME REQUIRED
Purgeable Halocarbons by GC (502.2, 601, 8010, 8021)	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	14 days	40mL
Purgeable Aromatic Hydrocarbons by GC (502.2, 602,8020,8021)	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	14 days	40mL
Halocarbons and Aromatics (502.2, 601/602, 8010/8020, 8021)	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	14 days	40mL
Acrolein & acrylonitrile (603)	3 x 40-ml G	none	3 days	40mL
VOC by 8015B-VOC	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	14 days	40mL
GRO/petroleum products by 8015B	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	14 days	40mL
Alcohols by 8015B (DAI)	3 x 40-ml G	none	14 days	40mL
Glycols by 8015B (DAI)	3 x 40-ml G	none	14 days	40mL
Solvents by 8015B (DAI)	3 x 40-ml G	none	14 days	40mL
Methanol and other Solvents(NCASI)	3 x 40-ml G	none	28 days	40mL
Dissolved Gases in water (FID)	3 x 40-ml G	0.3mL 1:1 hydrochloric acid	14 days	40mL
Dissolved Gases in water (TCD)	3 x 40-ml G	none	14 days	40mL

(1) Containers should be iced at time of collection in addition to the chemical preservation
(2) If the sample is chlorinated, sodium thiosulfate is added to the vials prior to shipment or is added the time of collection to destroy residual chlorine.
(3) The hold time for VOC is 7 days if the samples are not preserved with HCl at the time of collection

ANALYSES/CONTAINERS FOR LIQUIDS-VOC by GC/MS

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME (3)	MINIMUM VOLUME REQUIRED
Volatiles by GC/MS (524.2, 624, 8240/8260)	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	14 days	40mL
Volatiles by Isotope Dilution (1624)	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	14 days	40mL
Volatiles by CLP 3/90 and 10/92	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	10 days(4)	40mL

(1) Containers should be iced at time of collection in addition to the chemical preservation
(2) If the sample is chlorinated, sodium thiosulfate is added to the vials prior to shipment or is added the time of collection to destroy residual chlorine.
(3) The hold time for VOC is 7 days if the samples are not preserved with HCl at the time of collection
(4) The hold time for CLP methods is measured from the date of receipt into the laboratory.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS- VOC by VG/VM

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME (3)	MINIMUM WEIGHT REQUIRED
Purgeable Halocarbons by GC (8010, 8021)	2 x 5-g Encore plus 125-mL G amber	5mL 5% sodium bisulfate solution or frozen in water (2)	14 days 7days	1 5-g Encore plus 125-mL bulk
Purgeable Aromatic Hydrocarbons by GC (8020,8021)	2 x 5-g Encore plus 125-mL G amber	5mL 5% sodium bisulfate solution or frozen in water (2)	14 day 7 days	1 5-g Encore plus 125-mL bulk
Halocarbons and Aromatics (8010/8020, 8021)	2 x 5-g Encore plus 125-mL G amber	5mL 5% sodium bisulfate solution or frozen in water (2)	14 days 7 days	1 5-g Encore plus 125-mL bulk
GRO (8015B)	2 x 5-g Encore plus 125-mL G amber	5mL 5% sodium bisulfate solution or frozen in water (2)	14 days 7 days	1 5-g Encore plus 125-mL bulk
VPH (Ma Method)	2 x 25-g Encore	None	(5)	25g
Volatiles by GC/MS (8260)	2 x 5-g Encore plus 125-mL G amber	5mL 5% sodium bisulfate solution or frozen in water (2)	14 days 7 days	1 5-g Encore plus 125-mL bulk
Volatiles by GC/MS CLP 3/90	125-mL G amber	None(1)	10 days (4)	25g

(1) Containers should be iced at time of collection.
(2) The sample must be preserved with sodium bisulfate within 48 hours of collection if not preserved in the field. If the sample contains high levels of carbonates that prevents the use of sodium bisulfate, the sample may be frozen in reagent water within 48 hours of collection.
(3) The hold time for VOC is 48 hours if the samples are not preserved with sodium bisulfate or frozen in water within 48 hours of collection
(4) The hold time for CLP methods is measured from the date of receipt into the laboratory.
(5) Samples for VPH must be extracted with methanol within 48 hours of collection (1mL methanol per gram of sample). The extract must be analyzed within 28 days of collection.

ANALYSES/CONTAINERS FOR LIQUIDS-Semivolatiles by GC

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Microextractables (504/8011)	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (2)	28 days	40mL
Pesticides (505, 618)	3 x 40-ml G	none (2)	7 days	40mL
Phenols GC(604,8041)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Benzidines (625,8270C)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Phthalate esters (606/8061)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Nitrosamines(607/8071)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Chlorinated Pesticides (508, 608, 608.1, 608.2, 617, 645, 8080/8081)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Chlorinated pesticides and PCBs as Aroclors (8081/8082)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
PCBs (608/8080/8082) as Aroclors or Congeners	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Chlorinated pesticides and PCBs as Aroclors CLP 3/90 and 10/92	2 x 1-L amb G	none (2)	5 days/40 days (3,4)	1-L
Nitroaromatics & isophorone (609/8091)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
PAHs (610/8100)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Haloethers (611/8111)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Chlorinated hydrocarbons (612/8121)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Organophosphorous Pesticides (507, 614/614.1, 622/622.1, 8141)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Chlorinated Herbicides (515,615/8151)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Triazine Pesticides (619/620)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Nitroaniline Pesticides (627)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Organonitrogen Pesticides (633)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Thiocarbamate Pesticides (634)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Petroleum hydrocarbons/EPH/DRO (8015B)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L

(1) Containers should be iced at time of collection in addition to the chemical preservation
(2) If the sample is chlorinated, sodium thiosulfate is added to the vials prior to shipment or is added the time of collection to destroy residual chlorine.
(3) The first number is the hold time until the extraction; the second time is the hold time for the extract; i.e., the extraction must take place within 7 days of collection and the extract must be analyzed within 40 days of the date of extraction.
(4) The hold time for CLP methods is measured from the date of receipt into the laboratory.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-Semivolatiles by GC

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM WEIGHT REQUIRED
Phenols GC(8041)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Phthalate esters (8061)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Nitrosamines(8071)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Chlorinated/ Pesticides (/8080/8081)	250-ml or 500-mL G	none	14days/40 days (2)	30g
PCBs (8080/8081)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Chlorinated Pesticides and PCBs as Arclors CLP 3/90	250-ml or 500-mL G	none	10days/40 days (2,3)	30g
Nitroaromatics & isophorone (8091)	250-ml or 500-mL G	none	14days/40 days (2)	30g
PAHs (8100)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Haloethers (8111)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Chlorinated hydrocarbons(8121)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Organophosphorous Pesticides (8141)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Chlorinated Herbicides (8151)	250-ml or 500-mL G	none	14days/40 days (2)	30g
Petroleum hydrocarbons/EPH/ DRO (8015B)	250-ml or 500-mL G	none	14days/40 days (2)	30g

(1) Containers should be iced at time of collection in addition to the chemical preservation
(2) The first number is the hold time until the extraction; the second time is the hold time for the extract; i.e., the extraction must take place within 7 days of collection and the extract must be analyzed within 40 days of the date of extraction.
(3) The hold time for CLP methods is measured from the date of receipt into the laboratory.

ANALYSES/CONTAINERS FOR LIQUIDS-Semivolatiles by GC/MS

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Base/Neutrals/Acids by GC/MS (525,625/8270)	2 x 1-L amb G	none (2)	7days/40 days(3)	1-L
Base/Neutrals/Acids by GC/MS CLP 3/90 and 10/92	2 x 1-L amb G	none (2)	5 days/40 days(3,4)	1-L
Dioxins and Furans (1613/8280/8290)	2 x 1-L amb G	none (2)	30days/45 days(3)	1-L
2,3,7,8-TCDD (613)	2 x 1-L amb G	none (2)	7days/40 days(3)	1-L
PCB Congeners (680)	2 x 1-L amb G	none (2)	7days/40 days(3)	1-L
Chlorinated Phenolics (1653)	2 x 1-L amb G	2mL 1:1 sulfuric acid(2)	30days/30days(3)	1-L

(1) Containers should be iced at time of collection in addition to the chemical preservation
(2) If the sample is chlorinated, sodium thiosulfate or ascorbic acid is added to the vials prior to shipment or is added the time of collection to destroy residual chlorine.
(3) The first number is the hold time until the extraction; the second time is the hold time for the extract; i.e., the extraction must take place within 7 days of collection and the extract must be analyzed within 40 days of the date of extraction.
(4) The hold time for CLP methods is measured from the date of receipt into the laboratory.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-Semivolatiles by GC/MS

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM WEIGHT REQUIRED
Base/Neutrals/Acids by GC/MS (625/8270)	250-mL or 500-mL amb G	none	7days/40 days (2)	30g
Base/Neutrals/Acids by GC/MS CLP 3/90	250-mL or 500-mL amb G	none	10 days/40 days (2,3)	30g
Dioxins and Furans (8280)	250-mL or 500-mL amb G	none	30days/45days (2)	30g

(1) Containers should be iced at time of collection in addition to the chemical preservation
(2) The first number is the hold time until the extraction; the second time is the hold time for the extract; i.e., the extraction must take place within 7 days of collection and the extract must be analyzed within 40 days of the date of extraction.
(3) The hold time for CLP methods is measured from the date of receipt into the laboratory.

CONTAINERS FOR LIQUIDS-Liquid Chromatography

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
N-Methylcarbamoxyloximes & N-Methylcarbamates (531)	125-ml amb G	monochloroacetic acid to pH <3	28 days	10mL
Glyphosate (547)	125-ml amb G	None	14 days (18 months if frozen)	10mL
Endothall (548)	1-L amb G	None	7 days/ 14 days	100mL
Diquat and Paraquat (549.1)	500-mL high density ,foil wrapped PCB with Teflon-lined cap	sulfuric acid to pH<2	7days/21 days (3)	75mL
Hexachlorophene and Dichlorophene (604.1)	2 x 1-L amb G	None	7days / 40days	1-L
Cyanizine (629)	2x 1-L amb G	None	7 days/ 40 days	1-L
Benomyl (as Carbendazim) (631)	2 x 1-L amb G	None	7 days/ 40 days	1-L
Carbamate & Urea Pesticides (632)	2 x 1-L amb G	None	7 days/ 40 days	1-L
Rotenone (635)	2 x 1-L amb G	None	7 days/ 40 days	1-L
Bensulide (636)	2 x 1-L amb G	None	7 days/ 40 days	1-L
Oryzalin (638)	2 x 1-L amb G	None	7 days/ 40 days	1-L
Beniocarb (639)	2 x 1-L amb G	None	7 days/ 40 days	1-L
Bentazon (643)	2 x 1-L amb G	None	7 days/ 30 days	1-L
Picloram (644)	2 x 1-L amb G	None	7 days/ 30 days	1-L
PAHs (610/8310)	2 x 1-L amb G	none(2)	7 days/ 40 days(2)	1-L
Carbonyl Compounds (acetaldehyde and formaldehyde) (8315)	2 x 125 ml amb G	None	3 days/3 days	100mL
Acrylamide(8316)	125 ml amb G	None	7 days	10mL
N-Methylcarbamates (8318)	2 x 125 ml amb G	Monocloroacetic acid to pH <3	7 days/ 40 days	100mL
Nitroaromatics and Nitramines (explosive residues) (8330)	2 x 1-L amb G	None	7 days/ 40 days	1-L

CONTAINERS FOR LIQUIDS-Liquid Chromatography

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Acrylic acid (SL SOP)	40-mL G, Teflon septum	None	7 days/ 40 days	10mL
Asulam (SL SOP)	125-ml amb G	None	21 days	50mL
Cyanuric acid	125-ml amb G	None	Not specified	10mL
Ethylenthiourea (SL SOP)	125-ml amb G	None	7 days/40 days	10mL
Maleic acid/maleic anhydride (SL SOP)	40-mL G, Teflon septum	None	7 days/ 40 days	10mL
Phthalic acid/phthalic anhydride (SL SOP)	40-mL G, Teflon septum	None	7 days/ 40 days	10mL
Thiodiglycol (SL SOP)	125-ml amb G	None	7 days/ 40 days	10mL

CONTAINERS FOR SOILS-Liquid Chromatography

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Benomyl (as Carbendazim) (631)	100-mL G	none	14 days/ 40 days(2)	20g
Carbamate & Urea Pesticides (632)	100-mL G	none	14 days/ 40 days(2)	30g
Oryzalin (638)	100-mL G	none	14 days/ 40 days(2)	30g
Carbonyl Compounds (acetaldehyde and formaldehyde) (8315)	100-mL G	none	3 days/ 3 days(2)	20g
N-Methylcarbamates (8318)	100-mL G	none	7 days	10g
Nitroaromatics and Nitramines (explosive residues) (8330)	100-mL G	none	14 days	20g
Asulam (SL SOP)	100-mL G	none	14 days	2g
Cyanuric acid	100-mL G	none	Not specified	5g
Ethylenthiourea (SL SOP)	100-mL G	none	14 days	2g
Thiodiglycol (SL SOP)	100-mL G	none	14 days	2g
Acrylamide(8316)	100-mL G	none	14 days	2g
Acrylic acid (SL SOP)	100-mL G	none	14 days	2g
Maleic acid/maleic anhydride (SL SOP)	100-mL G	none	14 days	5g
Phthalic acid/phthalic anhydride (SL SOP)	100-mL G	none	14 days	5g

(1) Containers should be iced at time of collection in addition to the chemical preservation

(6) The first number is the hold time until the extraction; the second time is the hold time for the extract; i.e., the extraction must take place within 7 days of collection and the extract must be analyzed within 40 days of the date of extraction.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-Liquid Chromatography

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM WEIGHT REQUIRED
PAHs (8310)	2 x 1-L amb G	none	14days/40 days	30g
Carbonyl Compounds (formaldehyde)	2 x 125 ml amb G	none	3days/3days	10g

(1) Containers should be iced at time of collection.

CONTAINERS FOR LIQUIDS-Radiological Parameters

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME	MINIMUM VOLUME REQUIRED
Radionuclides, alpha	1-L P	nitric acid to pH<2	6 months	1-L P
Radionuclides, beta	1-L P	nitric acid to pH<2	6 months	1-L P
Radium (total as Radium 226)	4-L P	nitric acid to pH<2	6 months	4-L P
Radium 226	4-L P	nitric acid to pH<2	6 months	4-L P
Radium 228	4-L P	nitric acid to pH<2	6 months	4-L P
Tritium	1-L P	none	6 months	1-L P

ANALYSES/CONTAINERS FOR LIQUIDS-Microbiological

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Coliform, fecal and total (in drinking water)	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodium thiosulfate	30 hours	100mL
Coliform, fecal and total	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodium thiosulfate	6 hours	100mL
Fecal streptococci	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodium thiosulfate	6 hours	100mL
Bacteria plate count	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodium thiosulfate	6 hours	100mL
Chlorophyll	125-mL amb G	none	NA	125mL

(1) Containers should be iced at time of collection in addition to the chemical preservation

ANALYSES/CONTAINERS FOR HAZARDOUS WASTE PARAMETER TESTING

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM WEIGHT REQUIRED
TCLP	500-mL G	none	14 days(2)	100g(3)
SPLP	500-mL G	none	14 days(2)	100g(3)
Cyanide, reactive	250-mL P,G	none	14 days	10g
Sulfide, reactive	250-mL P,G	none	7 days	10g
Ignitability	250-mL G	none	NA	50mL/50g
Corrosivity (pH)	250-mL G	none	analyze ASAP	5g
Corrosivity (to steel))	250-mL G	none	NA	100mL/100g

(1) Containers should be iced at time of collection in addition to the chemical preservation
(7) The hold time is the maximum time until the leaching procedure is performed; after leaching, the routine liquid hold times apply.
(8) If the TCLP or SPLP samples is a liquid, a minimum volume of 1L should be requested to support the various analyses. The following are the minimum volumes of liquid sample or leachate to report the target analytes at the CQAP limits and below the regulatory threshold limits:

ANALYSIS	Minimum Volume of Leachate or liquid sample required
Base/neutrals/acids (8270)	200mL
Chlorinated pesticides (8081)	20mL
Chlorinated herbicides (8151)	20mL
Metals (6010)	20mL
Mercury (7470)	1.0mL
Volatiles (8260)	40mL

ANALYSES/CONTAINERS FOR WIPES

PARAMETER	ROUTINE MEDIA	WIPE SOLVENT	VIAL	MINIMUM REQUIRED
Chlorinated Pesticides and PCBs	2" x 2" gauze	Hexane	Glass	1 gauze
Chlorinated Herbicides	2" x 2" gauze	Methanol	Glass	1 gauze
Phenols	2" x 2" gauze	Methanol	Glass	1 gauze
Phthalate esters	2" x 2" gauze	Hexane	Glass	1 gauze
PAHs	2" x 2" gauze	Methanol	Glass	1 gauze
Explosives	2" x 2" gauze	Methanol	Glass	1 gauze
Volatiles	2" x 2" gauze	purge and trap methanol	Glass	1 gauze
Metals (except mercury)	2" x 2" gauze	Acetic acid solution	Plastic	1 gauze
Mercury	2" x 2" gauze	Acetic acid solution	Plastic	1 gauze

ANALYSES/CONTAINERS FOR AIR SAMPLES

PARAMETER	ROUTINE MEDIA(1)	PRESERVATIVE	HOLD TIME	MINIMUM VOLUME REQUIRED(3)
Volatiles				
TO1/TO2 VOC by Thermal Desorption	Sorbent Tube (Carbotrap 300)	None(2)	30 days	As received (1L max volume)(4)
TO14 VOC	Summa Canister	None (Do Not Ice)	30 days	As received
EPA18 (VOC and other gases collected in Tedlar Bags)	Tedlar Bag	None (Do Not Ice)	3 days	As received
EPA 18 VOC	"Shaving Cans"	None (Do Not Ice)	30 days	1 can
Semivolatiles				
TO4 Pesticides and PCBs (High Volume Sampler)	PUF	None(2)	7 days/40 days	As received
TO10-Pesticides and PCBs (Low Volume Sampler)	PUF	None(2)	7 days/40 days	As received
TO13-Selected BNA (High Volume Sampler)	PUF/Resin	None(2)	7 days/40 days	As received
TO13-Selected BNA (Low Volume Sampler)	PUF/Resin	None(2)	7 days/40 days	As received
NIOSH/OSHA				
Methanol	Silica Gel	None(2)	6 Weeks	1 liter
Acid anions-Cl, F, NO3, SO4	Silica Gel	None(2)	21 days	3 liters
VOC (Aromatics- NIOSH 1501/OSHA 07)	Charcoal	None(2)	2 weeks	5 liters
VOC (Halogenated-NIOSH 1003/ OSHA07)	Charcoal	None(2)	Not specified (14 days)	15 liters
PAH (NIOSH 5501)	Resin	None(2) Protect from heat and UV light	Not specified (14 days)	200 liters
Metals by ICP (NIOSH 7300/Osha ID-125)	Filter	None	Not specified (180 days)	1250 liters
Metals by GFAA	Filter	None	Not specified (180 days)	1250 liters

(1) Media may be purchased directly from the vendor
(2) No chemical preservative is required. Samples are iced at the time of collection and maintained at method-specified temperature until extraction and analysis.
(3) The quantitation limit will be calculated from the volume of sample supplied. The minimum volume for the NIOSH/OSHA methods is the minimum volume that can be sampled to meet the threshold limits. The maximum volume for a sorbent tube is 1.0L (1000mL). This is below "breakthrough" volume of the more volatile gases (e.g., vinyl chloride).

7.0 SAMPLE CUSTODY

7.1 Sample Custody Objectives

The primary objective of SL's sample chain-of-custody procedures is to provide accurate, verified, and traceable records of sample possession and handling from sample container shipment through laboratory receipt and sample disposition.

Evidence of documentation of sample collection, shipment, laboratory receipt and custody is accomplished utilizing a chain-of-custody record (Figure 7.1). A sample is considered in custody if it is:

- in actual possession of the sampler or transferee
- in view after being in physical possession of the sampler or transferee
- sealed so that sample integrity will be maintained while in possession of the sampler or transferee
- in a secured area, restricted to authorized personnel.

7.1.1 Custody Record Maintenance

Field and laboratory records, including copies of the chain-of-custody forms and associated field documentation, are maintained in a secure area with other project records. All field and laboratory data are reported in bound notebooks and entries are made in waterproof ink. Field and laboratory data entry errors are deleted with a one-line strike through the error. Correction tape or other substances designed to obliterate documentation are strictly prohibited in the laboratory or custody areas. The correction is initialed and dated by the sampling or analytical staff member making the change. Field and laboratory information is documented on prepared forms. All forms for recording field and laboratory data include spaces for data and initials which must be completed by the data recorder. Field and laboratory documentation not recorded on prepared forms is also dated and initialed

7.2 Sample Custody Procedures

All samples are received by the custody technician using custody procedures detailed in SL SOP CU01: *Receipt, Log Number Assignment, and Distribution of Field Samples*. The procedures for the preparation of sampling kits are described in SL SOP CU15: *Preparation of Sampling Containers*.

7.3 Laboratory and Field Custody Procedures

The following procedures apply to the custody activities observed by Savannah Laboratories during sample or legal custody procedures.

7.3.1 Selection and Preparation of Sample Containers Supplied to a Client of Sampling Team

Sample containers provided by SL are constructed from EPA-designated materials and contain EPA-prescribed preservatives. If requested, an SL (Figure 7.2) or client supplied identification label is affixed to the container. In order to monitor container temperature, a 100-mL plastic container labeled "Container Temperature- For Laboratory Use Only" is pre-filled with tap water and supplied with each sample shipment to monitor sample temperature upon receipt.

Pre-cleaned sample containers are purchased by Savannah Laboratories. Containers from each lot are pre-certified in-house prior to use in accordance with SL SOP CU35: *Procedure for Contaminant-Free Containers*.

7.3.2 Chain of Custody Documentation, Traceability, and Sample Integrity

Formal chain-of-custody procedures are initiated by a ***custody technician*** who is responsible for organization and relinquishment of sample containers to the client or field personnel.

All field information must be properly recorded on the chain-of-custody form. Proper completion of the form is the responsibility of the ***field sampling manager or client*** and is requested prior to the relinquishment of the samples. If the site location is different from the client address, the site location is recorded in the "Project Name" space on the chain-of-custody form, or on the right hand side of the form if additional space is required. The sample identities assigned in the field are recorded in the "Sample Identification" column. Common carriers may identify themselves by signing the "Relinquished By" space on the chain-of-custody form.

For samples transported from the field to the laboratory by common carrier, chain of custody is maintained. Completed custody forms must accompany each sealed cooler, and are placed in a plastic bag and taped to the inside lid of the cooler. At the client's request, coolers are sealed in the field with the SL Custody Seal (Figure 7.3) or custody tape by the field sampling team to ensure that tampering will be immediately evident. A unique identification number is recorded on the seal and accompanying chain-of-custody form with waterproof ink.

The ***custody technician*** is responsible for the inspection of shipping containers upon laboratory receipt for overall integrity and to ensure that the contents have not been altered or tampered with during transit. If tampering is apparent, the sample receipt custodian immediately contacts the assigned ***project manager*** who is responsible for client notification. Any problem or abnormality detected is documented on an Anomaly Report (Figure 7.4), which is completed by the sample custody technician. Any corrective action required by the client is also documented.

If shipping containers arrive intact, they are immediately opened by the custody technician in the receiving area, and the chain-of-custody form and temperature container removed for inspection. Container temperature upon receipt is documented in a bound sample registry (Figure 7.5), or if requested by the client, documented on the chain-of-custody form.

7.3.3 Field Custody

When sample collection is performed by Savannah Laboratories, the SL field sampling manager is responsible for ensuring that chain-of-custody procedures for all sampling events are properly documented. The custody forms and login procedures follow the protocol outlined in Section 7.3.

Prior to field sampling, it is preferable to place waterproof sample labels on each sample container and complete each sample label with as much information as possible in waterproof ink. Field sampling technicians are responsible for ensuring that labels are complete. Each sample is identified in the field by a unique alphanumeric designation on the label.

Adequate sample identification information included on each container label must be included on all field-generated records including: permanent field notebook, individual well log, groundwater elevation form, and chain-of-custody form. This field documentation demonstrates traceability of the containers and samples and links all ancillary records to specific sampling events.

Each sample is packed to ensure against leakage and breakage and to maintain individual sample integrity. All glass containers are secured individually with bubble wrap. All VOA sample vials are wrapped in bubble bags. Plastic bags are supplied by SL to hold ice necessary to maintained the samples at less than 6°C during transit. An attempt should be made by the field sampling team to precool samples to 4°C prior to packing the sample cooler for shipment. Additional information regarding sample containers can be found in Section 6.0 and the appropriate SL SOPs.

When applicable to the site, the following information is documented by the field technicians in the bound field notebook. This field documentation is reviewed, approved, and initialed by the field sampling manager prior to client submission.

Site location
Date/time of sampling
Sample identification (including specific location)
Sample sequence number
Site conditions
Weather conditions
Description of QC samples collected
Names of personnel/visitors
Sampling/purging equipment used
Field analysis data
Field documentation techniques
Well casing composition and diameter
Drilling/boring method
Drilling well type/name
Water table and well depth
Purge volume calculations
Volume of water purged
Date/time of purging
Analytical data to monitor stabilization of well
Use of fuel powered units
Plumbing/tap material construction
Purging flow rate
Purging time
Flow rate at sampling collection
Depth samples taken
Beginning/ending time for composite sampling
Depth soil samples taken
Soil sampling technique used
Type/description of drums
Phases sampled in drums

7.3.4 Sample Documentation, Identification, and Login

A sequential identification number is assigned by division to the project and recorded on the chain-of-custody form, on each sample container submitted with the project and in the bound Sample Registry. Accurate and complete sample documentation must be provided on the chain-of-custody form in order to log samples into the sample registry. The sample registry includes all information necessary to maintain chain of custody including laboratory ID, client (field) ID, and initials of the custody technician. Ancillary information such as sample collection date and requested analyses is transferred from the chain-of-custody form into the LIMS, and appears on the client project-specific acknowledgement.

Once the chain of custody is verified, the project identified by this unique number is logged into the computerized LIMS (Figure 12.1) to transfer the desired work order request to the laboratory. The custody technician checks each sample against the chain-of-custody form for discrepancies between information on the sample label and information provided on the chain-of-custody form. The custody technician also inspects all samples for leakage or obvious seal tampering (if provided). All samples are unpacked in a well ventilated sample receipt area. Face shields are available to each sample receipt staff member for use with any hazardous samples. Samples received in plastic containers which appear to be accumulating or evolving gas are treated cautiously and inspected under a chemical hood because they may contain toxic fumes or be of an explosive nature.

A space labeled "custody intact" provided on the chain-of-custody form is used to describe the sample condition upon receipt. A "Y" indicates no custody problem was identified and a "N" indicates samples or container integrity was compromised and client notification and corrective action is required. At client request, a "Cooler Receipt Form" (Figure 7.6) can be completed to document custodial concerns at sample login.

Discrepancies noted by the custody staff are transmitted to the project and sample manager and are resolved with the client prior to laboratory work assignment. Discrepancies are documented on the Anomaly Report. The project manager and the custody department staff should attempt to resolve custody discrepancies expeditiously to avoid holding time compromises. After a decision concerning a sample has been made, the project manager or sample manager makes an initialed note on the original custody form which states person notified, time, date, and resolution, if applicable. This information is also documented on the Anomaly Report. A faxed or hard copy of custodial resolutions or project order alterations should be secured from the client prior to work initiation. Copies of this documentation are mailed to the client and maintained in the client file.

7.3.5 Sample Preservation

After addition of the project sequential identification number, the samples are distributed to the appropriate laboratory section sample storage areas. Color-coded dots and unique sample bottle types correspond to specific analyses and are stored at designated sample storage areas throughout the laboratory sections. Bound sample storage temperature logs are maintained for all sample storage refrigerators to assure proper temperature maintenance throughout the analytical process.

The color code scheme for the various preservatives used in SL's sample containers is described in the Sample Container Request Form which is submitted to the client along with the sample containers. This two-sided form is shown in Figure 7.7 and 7.8.

When preservation is required, sample containers used by the SL field sampling team contain premeasured portions of preservatives. Preservatives are obtained prior to each sampling event from parent stocks assayed and maintained by the laboratory. The effectiveness of pH adjustment by addition of acid or base to the samples is checked after sampling by pouring a small amount of the preserved sample into a small specimen cup and testing with narrow range pH paper. Because of the risk of compromising sample integrity, VOA samples cannot be checked in the field.

All samples received by Savannah Laboratories are checked for proper pH adjustment by the appropriate preparation or analytical department as soon after receipt as possible. The pH of each sample is checked, documented, and adjusted, if necessary.

7.3.6 Sample Security, Accessibility, Distribution, and Tracking

Only authorized personnel are permitted within the laboratory areas where samples are stored. Sample storage areas are designed to segregate volatile and nonvolatile samples. Standards and extracts are also departmentally controlled and stored separately.

After sample registry login and verification, samples are relinquished from the receiving area to the appropriate sample analysis storage area. Transfer of samples from the sample receipt personnel to the department is documented on the Sample Internal Custody Log (Figure 7.9). Interdivisional sample custody is documented on the Remote Division Sample Internal Custody Log (Figure 7.10). Using LIMS-generated sample preparation worksheets for guidance, samples are extracted, digested, or distilled as appropriate. An example sample preparation log (Wet Chemistry Extraction Log) is shown in Figure 7.11. The extracts, digestates, or distillates are then transferred and relinquished to the appropriate analysis section, where analysis is performed. An example analysis log (Fluoride Analysis) is shown in Figure 7.12. An example of a department-specific tracking form is shown in Figure 7.13 (BNA Extract Custody).

For projects where in-laboratory custody records are required by the client, the project manager and custody department will coordinate the documentation of these records.

Sample holding times are tracked via the LIMS. Sample collection dates are routinely entered into the LIMS with all sample logins. This information allows holding times specific to each department analysis to be tracked by department managers, supervisors, chemists, and analysts through the use of daily status sheets, reference sheets, and preparation worksheets. Date analyzed is recorded via instrument outputs or analysis forms/logs when applicable as an integral part of the raw data. For projects in which reporting the analysis or preparation date is appropriate, the dates are entered into the LIMS.

7.3.7 Sample Disposition Documentation

Upon completion of analytical work, sample custody of unused sample portions, extracts, or digests is relinquished to a central secured storage area. Here the samples, digest, or extracts await disposal, which is performed with the assistance of the LIMS. The LIMS stores clients' specific disposal instructions, compiles results from the analyses of composited samples, prepares sample disposal lists, invoices for disposal and sample return costs, and provides a disposal record for all excess samples.

7.3.8 Interdivisional Custody

The laboratory director at each location monitors the sample load and turnaround time through LIMS-generated reports. If it appears that analysis demand will exceed capacity, samples may be transferred (provided client contracts or arrangements, project QA plans or certification limitations do not prohibit sample transfer) to another SL division to ensure that holding times and turnaround commitments are met. The procedures used by SL are described in SL SOP CU20: *Interlaboratory Sample Exchange* and are summarized below.

If samples are transferred to another division laboratory, full custody is maintained. A completed and signed fax of a Interdivisional Shipping Log (Figure 7.14) is sent to the receiving division custody department. Special LIMS determination codes specific to each laboratory location are utilized to enable the project manager and laboratory director to track sample progress and maintain chain of custody. Copies of the original chain-of-custody form (executed for interdivisional sample submittal), computerized LIMS order information (LOI), and extract or digest preparation logs pertinent to the project order accompany the samples or sample preparations. The accompanying documentation also includes dates of sample preparation and requested analyses. For projects where reporting the preparation or analysis dates/times are entered into the LIMS so that it appears on the final report.

7.4 Verification of Hard Copy Records

Data worksheets, data approval forms, and final reports are routinely printed for verification and signatures. Hard copies of final reports, field data, chain-of-custody forms, and ancillary documentation pertinent to the project will be stored in a secured storage area and placed chronologically within alphabetically arranged client files.

7.5 Facility Security Policy

All external doors are either visually monitored by SL staff or kept locked. Visitors are required to sign in and wear a visitor's badge during their visit and are accompanied at all times by an SL staff member when in the laboratory. Lockable refrigerators and storage cabinets are available for samples requiring this level of security.

Serial Number 51229

SL *SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC.*

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

☐ 5102 LaRoche Avenue, Savannah, GA 31404 Phone: (912) 354-7858 Fax: (912) 352-0165
☐ 2846 Industrial Plaza Drive, Tallahassee, FL 32301 Phone: (904) 878-3994 Fax: (904) 878-9504
☐ 414 SW 12th Avenue, Deerfield Beach, FL 33442 Phone: (954) 421-7400 Fax: (954) 421-2584
☐ 900 Lakeside Drive, Mobile, AL 36693 Phone: (334) 666-6633 Fax: (334) 666-6696
☐ 6712 Benjamin Road, Suite 100, Tampa, FL 33634 Phone: (813) 885-7427 Fax: (813) 885-7049
☐ 100 Alpha Drive, Suite 110, Destrehan, LA 70047 Phone: (504) 764-1100 Fax: (504) 725-1163

PROJECT REFERENCE	PROJECT NO	P.O. NUMBER

PROJECT LOC (State)	SAMPLER(s) NAME	PHONE / FAX

CLIENT NAME	CLIENT PROJECT MANAGER

CLIENT ADDRESS (CITY, STATE, ZIP)

MATRIX TYPE: AQUEOUS (WATER) / SOLID OR SEMISOLID / AIR / NONAQUEOUS LIQUID (oil, solvent, etc)

REQUIRED ANALYSES

PAGE OF

☐ STANDARD REPORT DELIVERY

☐ EXPEDITED REPORT DELIVERY (surcharge)

Date Due: _____

SAMPLE DATE	SAMPLE TIME	SL NO	SAMPLE IDENTIFICATION	AQUEOUS (WATER)	SOLID OR SEMISOLID	AIR	NONAQUEOUS LIQUID (oil, solvent, etc)	NUMBER OF CONTAINERS SUBMITTED	REMARKS

RELINQUISHED BY (SIGNATURE)	DATE	TIME	RELINQUISHED BY (SIGNATURE)	DATE	TIME	RELINQUISHED BY (SIGNATURE)	DATE	TIME
RECEIVED BY (SIGNATURE)	DATE	TIME	RECEIVED BY (SIGNATURE)	DATE	TIME	RECEIVED BY (SIGNATURE)	DATE	TIME

LABORATORY USE ONLY

RECEIVED FOR LABORATORY BY: (SIGNATURE)	DATE	TIME	CUSTODY INTACT	CUSTODY SEAL NO.	SL LOG NO.	LABORATORY REMARKS
			☐ YES ☐ NO			

FIGURE 7.1

FIGURE 7.2

Client _____

Sample ID _____

Location _____

Analysis _____

Preservative _____

Collection Date/Time _____

Collected By _____

FIGURE 7.3

SL *SAVANNAH LABORATORIES*
& ENVIRONMENTAL SERVICES, INC.

OFFICIAL SAMPLE SEAL

SAMPLE ID

SIGNATURE

SEAL NO. DATE TIME

SEAL BROKEN BY

DATE

FIGURE 7.4

ANOMALY REPORT

Date: _____ Log #: _____ Sample ID: _____ Client: _____

Dept: ___TX ___GE ___LC ___ME ___RA ___CU ___SG ___SM ___VG ___VM ___AI

Analysis: _____

Reported by: _____

Anomaly:

☐ Sample matrix is different than indicated by log-in.

Logged in as	*Best described as*
Water	*Water*
Soil	*Non-aqueous liquid*
Oil	*Soil*
	Sludge
	Oil
	Product
	Other _____

☐ Sample was received with inadequate preservation, and was preserved upon receipt.

☐ Sample received in an incompatible sample container. ____ *glass* ____ *plastic* ____ *other* _____

☐ MS/MSD failed while the LCS/LCSD passed criteria, for a drinking water parameter. Method indicates data flagging.

☐ Target analyte(s) detected in drinking water sample. (Describe below)

☐ Sample exhibits gross non-homogeneity. (Describe below)

☐ Insufficient sample received for analysis.

☐ Data flag may be needed. Discuss with DM/LM before reporting.

Other _____

Custody: *ALWAYS ATTACH A COPY OF COC WITH HIGHLIGHTED DEFICIENCY

☐ Sample description discrepancy between COC & Container

☐ Sample container breakage

☐ Cooler temp >6°C or frozen

☐ Sample received not listed on COC

☐ Custody seals broken

☐ Incomplete COC

☐ Sample container partially filled

☐ Improperly preserved sample

Comments:

Client Notified: ☐Yes ☐No

Contact:

Date:

Resolution:

Route to:

Division PM: _____

Other Div. PM: SL_____ ML_____ NL_____ FL_____ TL_____ BL_____

FAN038:04.09.97:3

SL SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES INC

SL SAMPLE REGISTRY

					AQUEOUS											NONAQUEOUS												
SL LOG NO.	TIME RECEIVED	DATE RECEIVED	DELIVERED BY	CLIENT ID	L n/m plastic	L n/m amber glass w/TFE	500 mL m/m plastic	500 mL m/m amber glass	250 mL m/m plastic	250 mL m/m nalgene	125 mL m/m amber glass w/FTE	100 mL m/m plastic	40 mL vial w/TFE			L w/m glass	L w/m plastic	500 mL w/m glass	500 mL w/m plastic	250 mL w/m plastic	125 mL m/m amber glass w/FTE	100 mL w/m glass	250 mL m/m glass	ADDITIONAL SAMPLE INFORMATION	CONTAINER TEMP.	NOTIFY CLIENT	LOGIN COMPUTER	DATE REPORTED

FIGURE 7.5

FIGURE 7.6

COOLER RECEIPT FORM	
Client:	Project:
SL Log #:	Date Received:
SL Cooler Receipt Custodian (Signature):	

Use other side of this form to note details concerning custodial discrepancies

		YES	NO
1	Did a shipping slip (air bill, etc.) accompany the cooler shipment?		
2	Were custody seals affixed to the outside of cooler? If YES, enter the following: Seal Identification (if provided):		
3	Were custody seals unbroken and intact at the date and time of arrival?		
4	Were custody papers completed properly (ink, signed, etc.)?		
5	Chain of custody associated with cooler receipt form.		
6	Was wet ice/blue ice used? (Circle which media)		
7	Cooler temperature upon receipt:		
8	Describe type of packing in cooler (vermiculite, bubble pack, etc.).		
9	Were sampling containers supplied by SL or client? (Circle which one)		
10	Did all bottles arrive intact and were labels in good condition?		
11	Did all bottle labels agree with custody papers?		
12	Were bubbles present in VOA samples?		
13	Was the project manager notified of any custody discrepancies or excursions?		
14	Was a custody excursion form completed and a copy provided to the project manager? If so. complete No. 15.		
15	Who was contacted? By whom: Date:		

FIGURE 7.7

SL SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC.

5102 LaRoche Avenue • Savannah, GA 31404 • (912) 354-7858 • Fax (912) 352-0165

Shipping Address: _____

Date of Shipment:

Method of Shipment:

Project Reference:

Project Site Location:

Phone No:

Account No:

SAMPLE CONTAINER REQUEST FORM

AQUEOUS												NONAQUEOUS							
																			PRESERVATIVES
L n/m plastic	L n/m amber glass w/TFE	500 mL m/m plastic	500 mL m/m amber glass	250 mL m/m plastic	250 mL m/m nalgene	125 mL m/m amber glass w/TFE	100 mL m/m plastic	40 mL vial w/TFE				EnCore Samplers -25g	EnCore Samplers - 5g	125 mL m/m amber glass w/TFE	250 mL w/m glass	250 mL w/m plastic	500 mL w/m plastic	500 mL w/m glass	Lab Pk Prep. by: _____ Lab Pk Checked by: _____ Quantity of Lab Pks. Shipped: _____ Proj Mgr: _____ Coordinator: _____ Comments: _____
																			NO. OF CONTAINERS SHIPPED
																			NO. OF CONTAINERS/SAMPLE
																			SET(S) OF TRIP BLANKS (3/SET)
																			ANALYSIS

It is the shipper's responsibility to ensure samples are maintained at the appropriate temperature during transit.

PRESERVATION COLOR CODE KEY

BLACK(BK)	Contains Monochloroacetic Acid. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
RED(R)	CAUTION! STRONG OXIDIZER! CONTAINS NITRIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
GREEN(G)	CAUTION! CONTAINS SULFURIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
BLUE(B)	CAUTION! STRONG CAUSTIC! CONTAINS SODIUM HYDROXIDE. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
WHITE(W)	No preservatives added.
ORANGE(O)	No preservatives added.
TAN(T)	Contains Zinc Acetate. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
YELLOW(Y)	Contains Sodium Thiosulfate.
LT.BLUE(LB)	CAUTION! CONTAINS HYDROCHLORIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.

DO NOT inhale vapors that may be caused from a chemical reaction between the preservative and sample. Collect sample in a well-ventilated area or use appropriate breathing apparatus. NEVER RINSE sample containers. If skin contact with preservatives occurs, flush exposed areas IMMEDIATELY.

FIGURE 7.8

GENERAL SAMPLING INSTRUCTIONS

DO NOT PRE-RINSE CONTAINERS. These containers have been specially prepared for specific analyses (See Preservation Color Code Key). Fill container to within 1" of capacity unless otherwise indicated, cap tightly, label and ice. Some requests require multiple containers to perform all analyses. (See Sample Request Form on reverse side.)

LITER PLASTIC	
No preservative n/m:	Physical Properties, Miscellaneous General (BOD)
Red n/m:	Radiological (Rad 226, Rad 228, alpha and beta)
LITER AMBER GLASS	
No preservative n/m:	Extractable Organics (BNAs, Pesticides/PCBs, Herbicides), Dioxins/Dibenzofurans
500 ML PLASTIC	
Blue n/m:	Cyanide
No preservative m/m:	Physical Properties, Miscellaneous, General
Red m/m:	Metals with Mercury
500 ML GLASS W/TFE	
Lt. Blue m/m:	Petroleum Hydrocarbons
Green m/m:	Oil and Grease
Green m/m (amber):	TOX. Fill to capacity.
No preservative w/m: (Nonaqueous)	All Organics (excluding Volatiles), Inorganics, Physical Properties, General
250 ML PLASTIC	
No preservative w/m: (Nonaqueous)	Inorganics, Physical Properties, General (single parameter)
No preservative m/m:	Physical Properties, Inorganics (nutrients), Hexavalent Chromium
Red m/m:	Metals without Mercury
Green m/m:	Nitrogen series, Phosphorus
Tan m/m:	Sulfide
250 ML NALGENE	
Yellow m/m:	Bacteriological (Coliform, Standard Plate Count) Sterile container - do not touch cap or container interior. Remove Faucet strainer and flush line prior to sample collection.
125 ML AMBER GLASS W/TFE	
Lt. Blue m/m:	TOC. Fill to capacity
Green n/m:	Total Recoverable Phenolics
No preservative m/m: (Nonaqueous)	Volatiles. Fill to capacity - no headspace.
120 ML PLASTIC	
No preservative m/m:	Physical Properties. Inorganics (single parameter)
Green m/m:	Nutrients. COD (single parameter)
125 ML GLASS	
No preservative w/m: (Nonaqueous)	Organics, Inorganics, Physical Properties, General (single parameter)
40 ML GLASS VIAL W/TFE	
Lt Blue n/m:	Volatiles (aromatics and/or Halogenated constituents). Fill vials until slightly overflowing with minimum aeration. Place septa W/TFE liner facing sample and seal with NO headspace.
Orange n/m:	EDB, Volatile Halocarbons. Fill as referenced above.
Yellow n/m:	Trihalomethanes (THM). Fill as referenced above.

Container Closure Key (n/m = narrow mouth, m/m = medium mouth, w/m = wide mouth)

CONTAINER SHIPPING INSTRUCTIONS

After sample collection, please check all custody forms and sample containers for discrepancies. Sign the custody form and seal in the enclosed plastic bag. To avoid container leakage during transit, additional plastic bags have been included in the shipment to contain ice for sample preservation. Please place these ice bags between the samples and secure the lab pack for shipment. Return lab packs to Savannah Laboratories & Environmental Services, Inc., 5102 LaRoche Avenue, Savannah, GA 31404. If you have any questions concerning containers shipped or acceptable field substitutions, please contact your project manager or sample coordinator for assistance at (912)354-7878 or FAX (912) 352-0165.

FIGURE 7.9

SAMPLE INTERNAL CUSTODY LOG

SL LOG NO._____

CLIENT:_____ **COURIER:**_____

 # COOLERS/CLIENT:_____ **TEMPERATURE**_____

GENERAL CONTAINER TYPE	P	#	METALS CONTAINER TYPE	P	#	VOLATILE CONTAINER TYPE	P	#	EXTRACTION CONTAINER TYPE	P	#
LIQUID			LIQUID			LIQUID			LIQUID		
L N/M PLASTIC			500 M/M PLASTIC			40 ML VIAL			L N/M AMB GLASS		
250 AMB GLASS			250 M/M PLASTIC			SOIL			250 M/M AMB GLASS		
500 M/M PLASTIC			100 M/M PLASTIC			ENCORE SPLERS/25g			500 M/M AM GLASS		
500 M/M AMB GLASS			SOIL			ENCORE SPLERS/5g			500 M/M PLASTIC		
250 N/M PLASTIC			L W/M PLASTIC			125 AMB W/M W/SEPTA			250 M/M PLASTIC		
250 N/M PLASTIC			500 W/M PLASTIC			125 AMB W/M GLASS			SOIL		
250 M/M NALGENE			250 W/M PLASTIC			AIR			L W/M GLASS		
125 M/M AMB GLASS			OTHER			TEDLAR BAG			500 W/M GLASS		
100 M/M PLASTIC						SUMMA CANS			250 M/M GLASS		
DO BOTTLE						VACUUM CANS			OTHER		
SOIL						TUBES					
250 M/M NALGENE						OTHER					
OTHER											
						PLEASE VERIFY					
PLEASE VERIFY	✓		PLEASE VERIFY	✓		TOTAL CONTAINERS	✓		PLEASE VERIFY	✓	
TOTAL CONTAINERS			TOTAL CONTAINERS						TOTAL CONTAINERS		

RELINQUISHED INFORMATION:

CUSTODY INITIAL/DATE _____ **CUSTODY** INITIAL/DATE _____ **CUSTODY** INITIAL/DATE _____ **CUSTODY** INITIAL/DATE _____

GENERAL INITIAL/DATE _____ **METALS** INITIAL/DATE _____ **VOLATILES** INITIAL/DATE _____ **EXTRACTION** INITIAL/DATE _____

MISC. BOTTLES STORED IN REFRIGERATOR FOR SUBCONTRACT/REMOTE TRANSFER:

_____

FIGURE 7.10

REMOTE DIVISION

SAMPLE INTERNAL CUSTODY LOG

SL DIVISION_____

 # COOLERS_____

TEMPERATURE(S)_____

LOG # FOR GENERAL LAB	#	LOG # FOR METALS LAB	#	LOG # FOR VOLATILES LAB	#	LOG # FOR EXTRACTION LAB	#
PLEASE VERIFY ✓		PLEASE VERIFY ✓		PLEASE VERIFY ✓		PLEASE VERIFY ✓	
TOTAL CONTAINERS		TOTAL CONTAINERS		TOTAL CONTAINERS		TOTAL CONTAINERS	

RELINQUISHED INFORMATION:

CUSTODY INITIAL/DATE	**CUSTODY** INITIAL/DATE	**CUSTODY** INITIAL/DATE	**CUSTODY** INITIAL/DATE
GENERAL INITIAL/DATE	**METALS** INITIAL/DATE	**VOLATILES** INITIAL/DATE	**EXTRACTION** INITIAL/DATE

FIGURE 7.11

WET CHEMISTRY EXTRACTION LOG

Prep Date:_____ Analyst:_____ LCS True Value_____

Chemistry:_____ Batch ID:_____ LCS Spike Source:_____

Method#:_____ Final Volume:_____ MS Spike True Value_____

Start Time:_____ End Time:_____ MS Spike Source_____

SL Log #	Sample Description	Sample Weight

FIGURE 7.12

FLUORIDE
Method 340.2

CALIBRATION CURVE		
mg/L	Log (mg/L)	mV
0.1	-1	
0.3	-0.522	
1	0	
2	0.301	
3	0.477	
10	1	
20	1.30	

Date:
Analyst:
Batch #:
Correlation Coefficient:
QC Check (Source/True Value) :
Accuracy Range:
Spike Level:
Rev/Date:

#	SL Log No.	Sample Description	Sample Dilution	Volume or Wet Weight	Sample Potential (mV)	Result (mg/L)	Percent Solids	Result (mg/kg dw)	% REC	% RPD

FIGURE 7.13

SEMIVOLATILE EXTRACT CUSTODY LOG

QE #	Total # of Extracts	Box ID	Analysis Required	Date Complete (Final Pro) Chk)	Date Relinquished from Extraction Department		Date Received		Instrument Group	Date Disposed
					Int	Date	Int	Date		

FFX005.06.14.93 0

FIGURE 7.14

CU20:03.31.98:1

INTERDIVISIONAL SHIPPING LOG
***FAX PRIOR TO SHIPMENT OF SAMPLES**

PM:_____

DIVISION SENT FROM: _____ TO: _____

DATE OF SHIPMENT:_____

METHOD OF SHIPMENT:_____

SL LOG NO._____ DUE:_____ LOGGED IN: __Y __N
_____# SAMPLES/MATRIX _____# CONTAINERS
ANALYSIS REQUIRED (DETS PREFERRED)_____
_____
CERTIFICATION REQUIRED __Y __N TYPE_____

SL LOG NO._____ DUE:_____ LOGGED IN: __Y __N
_____# SAMPLES/MATRIX _____# CONTAINERS
ANALYSIS REQUIRED (DETS PREFERRED)_____
_____
CERTIFICATION REQUIRED __Y __N TYPE_____

SL LOG NO._____ DUE:_____ LOGGED IN: __Y __N
_____# SAMPLES/MATRIX _____# CONTAINERS
ANALYSIS REQUIRED (DETS PREFERRED)_____
_____
CERTIFICATION REQUIRED __Y __N TYPE_____

NOTE: PLEASE PROVIDE DOCUMENTATION OF SPECIAL REQUIREMENTS FOR CERTIFICATION PRIOR TO ANALYSIS.

PM SIGNATURE/DATE:_____

RECEIVING DIVISION
SL LOG #'S:_____
RECEIPT INITIALS/DATE:_____
PROBLEMS __Y __N (IF YES, PLEASE DESCRIBE)_____
_____
FAX INITIALS/DATE:_____

8.0 ANALYTICAL PROCEDURES

The ultimate responsibility for analytical method selection lies with the client or regulatory agencies. Whenever possible, laboratory and field analysis of all samples is conducted by EPA approved methodology or guidance. Interpretation of ambiguous or conflicting method requirements is accomplished by consulting with regulatory agencies and EPA Laboratory/QA personnel. When EPA approved methods do not exist or project protocols require alternative methods or modifications of EPA methods (i.e., to achieve lower reporting limits), methods are modified based on scientific logic and regulatory alternative method guidance.

Tables 5.1 and 5.2 list Savannah Laboratories' routine laboratory parameters with their respective method numbers. Table 5.3 lists the parameters for TCLP samples. Tables 5.4 through 5.7 list parameters for air, biological tissue, wipes, and oily samples with their respective method numbers, and Table 5.8 lists field parameters.

For Gas Chromatographic (GC) methods, which have long lists of targets, have peaks that co-elute, or are subject to matrix interferences (i.e., Methods 8021, 604/8041, 606/8061, 607/8070, 609/8091, 610/8100, 612/8121), SL recommends the guidance in SW846, which states that GC-Mass Spec (GC/MS) methods are preferred, provided project reporting detection limits are met and costs are not appreciably different. This practice will usually result in fewer false positive detects and more accurate results (usually lower due to the ability by GC/MS to separate unresolved peaks or eliminate matrix interferences).

A detailed SOP has been prepared for each routine analytical method. At a minimum, the SOPs are reviewed annually and updated as needed to include newly promulgated methods. If multiple versions of a reference method exist (e.g., 6010A, 6010B, etc.), the SOP will reflect the different requirements of each version or an SOP will be prepared for each version. Any modifications to the approved methodology are described in the SOPs. Copies of the SOPs are approved by laboratory management and issued under document control. A master copy of each SOP is retained by the laboratory's QA Department and are made available to each staff member involved in the procedure.

When new routine analytes are added to an established procedure or when a new routine method is developed, data for the analyte or method are generated, reviewed, and documented in accordance with SL SOP AN52: *Test Procedure for Method Development and Modification.* The main elements of AN02 are:

- -Preparation or modification of an SOP, if required (SL SOP AN01)
- -Performance and evaluation of the Initial Demonstration of Capability (SL SOP CA92)
- -Performance of an MDL Study (SL SOP CA90)

8.1 Laboratory Glassware

8.1.1 Volumetric Glassware

Savannah Laboratories employs appropriate glassware for all preparatory and analytical operations. For critical measurements, such as standard preparation, Class A volumetric glassware is used, when practical. Exceptions include the use of volumetric syringes for volatiles standards preparation and polypropylene volumetric flasks for metals standards.

8.1.2 Glassware Cleaning Procedures

Laboratory glassware washing procedures are adapted from SW-846, *40 CFR Part 136, Standard Methods*, and EPA 600/4-79-109. The procedures are given in SL SOP AN60:*Glassware Cleaning Procedures* and are summarized as follows:

Extractable Organics

The glassware is washed with hot water and a non-phosphate detergent. The glassware is scrubbed vigorously with a brush to remove all artifacts and rinsed three (3) times with tap water. The glassware is allowed to air dry whenever possible and stored inverted or with cap openings covered with aluminum foil or glass stoppers to exclude dust and other contaminants. Whenever possible, precleaned, certified, disposable glassware is utilized for extraction and extract storage.

Volatile Organics

The glassware is washed with tap water and nonphosphate detergent, rinsed thoroughly with organic free water, and oven dried at 110°C-120°C for at least two hours. Class A volumetric glassware is air dried. Glassware is usually stored in the oven until use. Caps and septa are washed in the same manner, but caps are air dried. Highly contaminated glassware is allowed to soak in Nochromix solution overnight, then washed as above.

General Chemistry, Microbiology, Nutrients, Demands

The glassware is washed with hot tap water and nonphosphate detergent, rinsed thoroughly with tap and deionized water, air dried, and stored inverted or foil placed over cap openings. Bateriological laboratory glassware and collection bottles are autoclave as described in analytical procedures or purchased presterlized (disposable). COD digestion tubes and caps are cleaned with brushing and tap water (no soap) and rinsed thoroughly with deionized water. Tubes for TKN and total phosphorus sample digestions are washed with hot water and phosphate free detergent, and rinsed with tap water, Nochromix, and deionized water. Whenever possible, precleaned, certified disposable glassware is utilized.

Metals/Radionuclides

The glassware, plastic, and Teflon items are washed in hot tap water and phosphate-free detergent. They are then rinsed with tap water, 1:1 nitric acid, tap water, and deionized water. For highly contaminated samples, it is recommended that Teflon beakers used for sample digestion are further decontaminated by adding 20mL nitric acid and 12mL hydrochloric acid, covered with a watch glass, and digested on a hot plate for two hours. Following this treatment, they are rinsed with 10% nitric acid and deionized water and allowed to air dry. Whenever possible, precleaned, certified disposable glassware is utilized for digestion and digestate storage.

8.2 Soil Sample Preparation Notes

In the absence of an approved soil method, water methods are adapted for soil matrices. The following soil preparation procedures are applied to parameters in Table 5.2.

1. Fluoride (extractable): Method 340.2/300.0

Approximately 5 g of sample is weighed out exactly and placed in a screw-cap bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle, the supernatant decanted, and the extract is analyzed as a liquid sample.

2. Gross Alpha and Gross Beta Particle Activity: Method 9310

Soil is ground to a fine powder with mortar and pestle, and 50 to 100mg soil is weighed onto a tared planchet. Sample is distributed evenly over planchet surface, fixed with clear acrylic solution, dried, and counted.

3. Chloride (extractable): Method 9251/9252/4500-CL⁻C/300.0

Approximately 5 g of sample is weighed out exactly and placed in a screw-cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle and the supernatant is decanted. The extract is analyzed as a liquid sample.

4. Sulfate (extractable): Method 9036/9038/375.3/300.0

Approximately 5 g of sample is weighed out exactly and placed in a 100-ml screw cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the extract is filtered using a syringe and filter with a 0.20-um pore size filter and analyzed as a liquid sample.

5. Orthophosphate (extractable): Method 365.1/300.0

Approximately 5 g of sample is weight out exactly and placed in a screw-cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped and placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle and the supernatant us decanted. The extract is analyzed as a liquid sample.

6. Benomyl in Soil: Method 631

Ten grams of sample are extracted and hydrolyzed in 2:1 methylene chloride:acidified methanol. The extract is cleaned by passing through a 5 gram alumina solid phase extraction cartridge. The cleaned extract is then concentrated, filtered, and analyzed as a liquid sample.

7. Acrylamide in Soil: Method 8316

Samples are prepared by adding 10 ml of HPLC grade H2O to 2.0 grams of sample in a 20 mL scintillation vial and sonicating for one hour. The leachate is then passes through a C18 solid phase extraction cartridge. The extract is filtered and analyzed as a liquid.

8.3 Validated Compounds and Modifications of Referenced Analytical Methods

Except for the instances described below, parameters in Tables 5.1 and 5.2 have been determined by the methods referenced with no significant modifications to those methods, other than the use of additional standards for parameters not included in the referenced method lists.

Asulam

Water samples are prepared by adjusting the pH to 3, saturating with NaCl and extracting with 1:1 acetonitrile:ethyl acetate. The extract in injected into the HPLC system following concentration. The HPLC system consists of an isocratic pump and a UV detector.

Thiodiglycol

Water samples are prepared by passing them through a solid phase cartridge. Soil samples are extracted by sonication with calcium chloride solution and then processed through the same cleanup as the water samples. The samples are injected into the HPLC system which consists of an isocratic pump and a UV detector.

Ethylenethiourea

Water samples are analyzed by passing them through a solid phase cartridge. Soil samples are extracted by sonication with water, then processed through the same cleanup as water samples. The samples are injected into the HPLC system which consists of an isocratic pump and a UV detector.

Nitrocellulose

Water samples are filtered through an inorganic membrane filter to isolate the nitrocellulose fibers which are suspended in the sample. The filter is then washed with methanol to remove any sources of organic nitrogen compounds while the nitrocellulose fibers remain on the filter. The fibers are dissolved in acetone, followed by evaporation of the acetone and subsequent hydrolysis of the residue in the presence of sodium hydroxide. Following neutralization, the sample is analyzed for nitrate and nitrite by a cadmium reduction spectrophotometric method or ion chromatography. The mass of nitrogen (as nitrate-nitrite) is used to calculate the concentration of nitrocellulose.

Soil samples are treated with a methanol rinse to remove any interfering sources of nitrogen. Acetone is then added to the soil to dissolve the nitrocellulose fibers. Following the addition of acetone, the sample is sonicated for about 10 hours. Centrifugation is then employed to separate the acetone from the soil residue. The soil residue is then treated and analyzed using the technique described above for water samples.

Phthalic acid/Phthalic anhydride and Maleic acid/Maleic anhydride

Soil samples are extracted by tumbling with reagent water. Soil sample extracts and water samples are adjusted to neutral pH and then cleaned by passing through C8 and amino solid phase extraction cartridges connected in series. Analysis is by HPLC with UV detection. All results are reported as the acid form.

Cryomazine

Cryomazine is extracted from soil samples by sonication with HPLC extraction fluid, consisting of hexanesulfonic acid, triethylamine, ammonium hydroxide, and phosphoric acid. Extract cleanup is accomplished using a C18 solid phase extraction cartridge, followed by HPLC with UV detection analysis.

Resorcinol

Water samples are acidified with phosphoric acid, then passed through a C18 solid phase extraction cartridge and filtered. Analysis is by HPLC using UV detection.

Arsenic in Water by Method 7060 (3020)

Water samples are prepared using the procedures in Section 7 of Method 3020. Arsenic is then determined using the analytical procedures described in Method 7060.

Selenium in Water by Method 7740 (3020)

Water samples are prepared using the procedures described in Section 7 of Method 3020. Arsenic is then determined using the analytical procedures described in Method 7740.

8.4 Reagent Storage and Documentation

Reagents are stored with consideration for safety and maximum shelf life. Storage conditions and documentation maintenance status for various classes of reagents are given in Table 8.1, as well as discussed below.

All acids, except those poured up in small marked containers which are for immediate use, are stored in the original containers in acid storage cabinets.

All bases, except those poured up in small containers for immediate use and those that are standardized for specific purposes, are stored in the original containers within designated areas or storage cabinets.

All flammable solvents, except those poured up for immediate use, are stored in original containers in approved vented flammable storage cabinets which are located indoors.

Dry reagents are stored in designated cabinets in cool, dry areas. Reactive chemicals, cyanides and sulfides are labeled and isolated from other chemicals.

All acids used for metal sample digestions and solvents used for semivolatile sample extraction are tested prior to initial use. Specific acceptable chemical lots are reserved and stored by the vendor(s) and are requisitioned and received as needed by the laboratory. Lot numbers used for digestion or extractions are recorded in bound notebooks in the appropriate departments.

Reagent blanks are analyzed with each sample batch for all methods, validating the purity of all reagents. All reagent containers are dated when received, and dated and initialed when opened (except high use items consumed in less than one week). Documentation is maintained to provide traceability of the reagents used with the analysis of any batch to specific reagent lot numbers.

8.5 Waste Disposal

Savannah Laboratories' divisions operate as either conditionally exempt small quantity generators of small quantity generators of hazardous waste.

All waste disposal is carried out in accordance with Savannah Laboratories' Waste Disposal SOP (CA70). This document includes procedures for identification, storage, personnel training, tracking forms, report forms, safety, as well as details of the disposal.

8.6 Sample Disposal

All waste disposal is carried out in accordance with Savannah Laboratories' Waste Disposal SOP (CA70). This document includes procedures for identification, storage, personnel training, tracking forms, report forms, safety, as well as details of the disposal.

After analysis completion, unused sample portions, extracts, or digests are transferred to a central secured storage area to await disposal. Unless a client requests the project manager to save unused samples, digests, or extracts, disposal from the central storage occurs as soon as holding times have expired or three weeks after results submission.

Requests for extended sample, digest, or extract storage must be provided by the client to the SL project manager in writing (or contract form) prior to sample receipt and extended storage may result in additional fees to be negotiated by the SL project manager prior to sample receipt. SL is not responsible for evaporation or other deterioration of samples, extracts, or digests during extended storage periods.

Samples which are requested to be returned to the client may be picked up at the laboratory by the client, shipped by courier (at the client's expense for packaged shipping) or returned by any other legal means that is arranged by the client. Clients requesting the return of samples should provide detailed shipping instructions.

If a client by contract requires that samples be disposed of by a hazardous waste contractor, the client's name and EPA ID number are used on the manifest and the client is billed for all disposal related costs.

9.0 CALIBRATION PROCEDURES AND FREQUENCY

9.1 Laboratory Equipment

Savannah Laboratories is equipped with state-of-the-art instrumentation to provide quality analytical data to clients. A list of the instrumentation maintained by Savannah Laboratories for the determination of the parameters contained in Tables 5.1, 5.2, 5.3, and 5.4 is found in Table 9.1. A list of all field instrumentation maintained by the laboratory is contained in Table 9.2.

9.2 Standard Receipt and Traceability

Standards are purchased from commercial sources in stock solutions or mixes designed for the specific methods or as neat analytes. Certificates of analysis are shipped with each standard material by the vendor. When possible, standards are certified to meet or exceed the criteria established by the US EPA or are traceable to NIST standards.

Standard traceability logbooks are maintained by all sections of the laboratory to track the receipt, preparation, and disposition of all standard materials. A lot number is assigned to each standard material and the lot number is documented in the standard traceability logbook along with date of preparation, initials of analyst, concentration, expiration date (if applicable), and solvent (if applicable). If required, a standard preparation narrative is also provided in this logbook to document the preparation steps for each stock standard.

9.3 Standard Sources and Preparation

Savannah Laboratories maintains an inventory of standard materials necessary to calibrate and verify all analytical systems. Table 9.3 lists the standard sources and preparation protocols for various sections of the laboratory. Field instruments requiring calibration standards (conductivity meters and pH meters) use the same sources as laboratory instrumentation. Table 9.4 lists titrants used by the laboratory and information regarding their standardization. Documentation of standard preparation is performed in accordance with SL SOP AN41: *Standard Material Traceability*. This SOP also provides guidance for the extending the expiration date of standards.

9.4 Laboratory Instrument Calibration

The calibration procedures for the various analytical methods are listed below. All CLP protocols are followed as written in the statement of work (SOW).

9.4.1 Metals

Metals are analyzed by three protocols: 200-series are primarily for drinking water and for NPDES compliance; 6000 and 7000-series are primarily for RCRA testing; and CLP protocols are primarily for hazardous waste site monitoring. It should be noted that EPA has promulgated two versions of EPA method 200.7; one for drinking water and the other for NPDES compliance testing.

ICP (Inductively Coupled Plasma-Atomic Emission)

The inductively coupled plasma atomic emission spectrophotometer is standardized daily with a single concentration standard solution containing the metals of interest and a calibration blank. Multi-point calibrations with a minimum of three standards and a calibration blank encompassing the concentration range of interest are analyzed annually and are on file for each ICP. The calibration curve demonstrates the linearity of each metal over the standardization range. Continuing calibration verification (CCV) standards are analyzed after every 10 samples and at the end of the sequence and must meet the acceptance criteria. A calibration blank (ICB or CCB) is analyzed immediately after the verification standards and must meet the acceptance criteria.

After the instrument is standardized, the following calibration verification checks are performed:

Calibration Check	6010	200.7 (NPDES)	200.7 (Drinking Water)
Re-analysis of Standards	+/-5% of true value	+/-5% of true value	+/-5% of true value
Initial Calibration Verification (ICV)	+/-10% of true value	+/-5% of true value	+/-5% of true value
Initial Calibration Blank(ICB)	<RL (<MDL)	<RL (<MDL)	<RL (<MDL)
RL Standard	Detected; +/-50% of true value	Detected; +/-50% of true value	Detected; +/-50% of true value
Interference Check Solutions A and AB	+/-20% of true value	+/-20% of true value	+/-20% of true value
Continuing Calibration Verification (CCV)	+/-10% of true value	+/-5% of true value	+/-10% of true value
Continuing Calibration Blank (CCB)	<RL (<MDL)	<RL (<MDL)	<RL (<MDL)

All sample results must be bracketed by acceptable calibration standards.

AA (Atomic absorption)

Atomic absorption spectrophotometers are calibrated daily with the specified number of calibration standards, including a calibration blank. The correlation coefficient of the regression curve must be greater than or equal to 0.995. An initial calibration verification (ICV) standard is analyzed immediately upon calibration and must meet acceptance criteria. Continuing calibration verification (CCV) standards are analyzed after every 10 samples and at the end of the sequence and must meet the acceptance criteria. A calibration blank (ICB or CCB) is analyzed immediately after the verification standards and must meet the acceptance criteria.

GFAA (Graphite furnace atomic absorption)

Calibration Check	7000-series	200-series	200.9
Minimum number of calibration points	4	4	4
Initial Calibration Verification (ICV)	+/-10% of true value	+/-10% of true value	+/-5% of true value
Initial Calibration Blank(ICB)	<RL (<MDL)	<RL (<MDL)	<RL (<MDL)
Continuing Calibration Verification (CCV)	+/-20% of true value	+/-10% of true value	+/-10% of true value
Continuing Calibration Blank (CCB)	<RL (<MDL)	<RL (<MDL)	<RL (<MDL)

All sample results must be bracketed by acceptable calibration standards.

FLAA (Flame atomic absorption)

Calibration Check	7000-series	200-series
Minimum number of calibration points	4	4
Initial Calibration Verification (ICV)	+/-10% of true value	+/-10% of true value
Initial Calibration Blank(ICB)	<RL (<MDL)	<RL (<MDL)
Continuing Calibration Verification (CCV)	+/-20% of true value	+/-10% of true value
Continuing Calibration Blank (CCB)	<RL (<MDL)	<RL (<MDL)

All sample results must be bracketed by acceptable calibration standards.

CVAA (Cold vapor atomic absorption-Mercury)

Calibration Check	7000-series	200-series
Minimum number of calibration points	6	4
Initial Calibration Verification (ICV)	+/-10% of true value	+/-5% of true value
Initial Calibration Blank(ICB)	<RL (<MDL)	<RL (<MDL)
Continuing Calibration Verification (CCV)	+/-20% of true value	+/-10% of true value
Continuing Calibration Blank (CCB)	<RL (<MDL)	<RL (<MDL)

All sample results must be bracketed by acceptable calibration standards.

9.4.2 General Chemistry

Autoanalyzer

The autoanalyzer is calibrated with a minimum of five calibration standards at least every six months.(some procedures/instruments may require daily calibration). The correlation coefficient of the curve must be greater than or equal to 0.995 using a regression fit. Independent calibration verification standards are analyzed immediately following the calibration standards (initial or continuing), after every 10 samples, and at the end of each run. Sample analyses must be bracketed by calibration verification standards that meet control criteria. The calibration curve is verified each day that analyses are performed by the analysis of a midpoint standard and by the analysis of a standard at the reporting limit of the target analyte(s). The standard at the midpoint must be ±10% of the true value of the standard and the standard at the reporting limit must be detected.

Ion Chromatography (IC)

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target analytes with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. Either linear regression or quadratic curve fitting is used, depending on the analyte. The regression correlation coefficient must be greater than or equal to 0.99 for any analyte to be used for quantitation. Calibration verification standards are analyzed immediately upon calibration, after every 10 samples, and at the end of each run. Sample analyses must be bracketed by calibration verification standards that meet the acceptance criteria.

Ultraviolet-Visible (UV-VIS) Spectrophotometer

The spectrophotometer is calibrated with a minimum of five standards at least every six months (some procedures/instruments may require daily calibration). The correlation coefficient of the curve must be greater than or equal to 0.995 using a regression fit. Independent calibration verification standards are analyzed immediately following the calibration standards (initial or continuing), after every 10 samples, and at the end of each run. Data must be bracketed by calibration verification standards that meet control criteria. The calibration curve is verified each day that analyses are performed by the analysis of a standard at the midpoint of the calibration curve and by the analysis of a standard at the reporting limit of the target analyte(s). The standard at the midpoint must be ±10% of the true value of the standard and the standard at the reporting limit must be detected.

Infrared (IR) Spectrophotometer

The infrared spectrophotometer is calibrated with a minimum of five standards at least every six months (some procedures/instruments may require daily calibration). The correlation coefficient of the curve must be greater than or equal to 0.995 using a regression fit. Independent calibration verification standards are analyzed immediately following the calibration standards (initial or continuing), after every 10 samples, and at the end of each run. Data must be bracketed by calibration verification standards that meet control criteria. The calibration curve is verified each day that analyses are performed by the analysis of a standard at the midpoint of the calibration curve and by the analysis of a standard at the reporting limit of the target analyte(s). The standard at the midpoint must be =10% of the true value of the standard and the standard at the reporting limit must be detected.

Turbidimeter

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. Gelex solid standards are calibrated against formazin standards initially and then quarterly. The instrument is calibrated daily with one Gelex standard for each range of interest. A mid-range calibration verification standard is analyzed for every 10 samples and must meet control criteria in order for bracketed data to be acceptable.

Conductivity Meter

The cell constant of each meter is determined at a minimum annually by the analysis of five KCl standards. To verify the cell constant, a verification standard is analyzed at the beginning of each working day, using a KCl standard in the expected range of the samples. For meters not having automatic temperature compensation, all samples are analyzed at 25 C ± 2 C.

pH Meter

The pH meter is calibrated daily with two standard buffers at pH 7.0 and either 4.0 or 10.0, and checked with a third buffer at 10.0 or 4.0, which must indicate ± 0.10 pH units of its given value. A calibration verification standard is analyzed immediately upon calibration and after every 10 samples. Acceptable calibration verification standards must bracket the sample analyses. Manual or automatic temperature compensation is performed, depending on the meter. Additional checks of the pH meter must be performed with buffers other than 4 or 10 if samples are outside the pH range of 4-10.

Total Organic Carbon (TOC)

The instrument is calibrated according to the manufacturer's recommendations, with a minimum of a single point calibration daily. A calibration verification standard is analyzed immediately upon calibration, after 10 samples, and at the end of each run. Sample analyses must be bracketed by acceptable calibration verification standards.

Ion Selective Electrode (ISE)

Ion selective electrodes are calibrated daily with a minimum of five standards. The calibration curve is established by linear regression applied to the log of the standard concentrations versus potential and must result in a correlation coefficient greater than or equal to 0.995. Calibration verification standards are analyzed immediately upon calibration, after every 10 samples, and at the end of each run. Data must be bracketed by calibration standards that meet control criteria to be acceptable.

Total Organic Halogens (TOX) /Absorbable Organic Halides (AOX)

Although the TOX/AOX instrument provides an "absolute" measurement of halogen, a six-point calibration curve (five point and a calibration blank) is analyzed to confirm the accuracy of the instrument readout. The coefficient of variation (percent relative deviation) must be less than or equal to 20% to confirm the validity of the calibration curve. The TOX/AOX calibration is verified by periodic analysis of a precision and recovery (PAR) standard, a mid-level calibration check standard.

Bomb Calorimeter

The energy equivalent of the bomb calorimeter is determined quarterly by bombing six standard benzoic acid tablets. A control standard is analyzed in duplicate for every batch of samples, and must meet control criteria in order for data to be acceptable.

Dissolved Oxygen (DO) Meter

DO meters are calibrated prior to use either by Winkler titration or the air calibration technique.

Temperature

All laboratory and field thermometers are calibrated annually by comparison with a NIST-certified thermometer. Field meters with automated temperature compensation are checked before use with a calibrated thermometer.

9.4.3 Gas Chromatography (GC)

Volatiles by GC (VG)

Volatile organic compounds (VOC) are analyzed by three GC protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; and 8000-series are primarily for RCRA testing. These analyses are generally performed using internal standard calibration and quantitation; therefore relative retention time, as defined in the respective SOPs will be used to determine the identification of the target compounds and bracketing by CCV will not be required unless specified in the method or QAPP. If external standard calibration is used, the absolute retention time window is calculated as three times the standard deviation obtained from a 72-hour sequence or default windows of 0.05 to 0.10 minutes are used for compounds where the calculated window is too restrictive or zero. Bracketing by CCV will be required for external standard calibrations if specified in the method, SOP, or QAPP.

Initial calibration (ICAL) is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard (CCV) must be analyzed periodically as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Calibration Check	500-series(1)	600-series	8000-series
Initial calibration -minimum calibration standards	3 (as the calibration range is extended, the number of points must be increased)	3	5
%RSD criteria (2)	<=20%	<=10%	<=20% (with exceptions noted below)
CCV criteria (%difference or %drift)	+/- 20%	Within Q-table values	+/-15% (with exceptions noted below)
Frequency of CCV	Every 8 hours	Daily	Every 12 hours

(1) An alternate single point calibration can be performed if the standard response is within 20% of the sample response.
(2) Alternatively, a regression curve (linear, quadratic, etc.) may be constructed. If the correlation coefficient of the regression curve is greater than or equal to 0.99, the curve may be used for quantitation of samples.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

8000-series CCV grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Semivolatiles by GC (SG)
Semivolatile organic compounds (SVOC) are analyzed by four GC protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; 8000-series are primarily for RCRA testing; and the CLP protocols are used for hazardous waste site monitoring. If internal standard calibration is used; relative retention time, as defined in the respective SOPs, will be used to determine the identification of the target compounds and bracketing by CCV will not be required unless specified in the method or QAPP. If external standard calibration is used, the absolute retention time window is calculated as three times the standard deviation obtained from a 72-hour sequence or default windows of 0.05 to 0.10 minutes are used for compounds where the calculated window is too restrictive or zero. Bracketing by CCV will be required for external standard calibrations if specified in the method, SOP, or QAPP.

Initial calibration (ICAL) is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD is less than or equal to the aceeptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard (CCV) must be analyzed periodically as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Calibration Check	500-series	600-series	8000-series
Initial calibration -minimum calibration standards	3 (as the calibration range is extended, the number of points must be increased)(1)	3	5
%RSD criteria(2)	<=20%	<=10%	<=20% (with exceptions noted below)
CCV criteria (%difference or %drift)	+/- 20%	+/-10%	+/-15% (with exceptions noted below)
Frequency of CCV	Every 8 hours	Daily	Every 12 hours

(1) An alternate single point calibration can be performed if the standard response is within 20% of the sample response.
(2) Alternatively, a regression curve may be constructed. If the correlation coefficient of the regression curve is greater than or equal to 0.99, the curve may be used for quantitation of samples.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

9.4.4 Gas Chromatography/Mass Spectrometry (GC/MS)

Volatiles by GC/MS (VM)
Volatile organic compounds (VOC) are analyzed by three GC protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; 8000-series are primarily for RCRA testing; and the CLP protocols are used for hazard waste site monitoring.

Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable EPA methods. Ion abundance acceptance criteria for VOC tuning with BFB are given below. Mass calibration is performed as an integral part of tuning.

The tune check and calibration check must be performed in the following intervals:

624 - Daily
8260/CLP - every 12 hours.
524.2 - every 8 hours.

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)			
	Ion Abundance Criteria		
m/e	524.2	624	8260/OLMO4.0(1)
50	15-40% of mass 95	15-40% of mass 95	8.0-40.0% of mass 95
75	30-80% of mass 95	30-60% of mass 95	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance	Base peak, 100% relative abundance	Base peak, 100% relative abundance
96	5-9% of mass 95	5-9% of mass 95	5.0-9.0% of mass 95
173	< 2% of mass 174	<2% of mass 174	< 2.0% of mass 174
174	> 50% of mass 95	> 50% of mass 95	50-120%% of mass 95
175	5-9% of mass 174	5-9% of mass 174	4.0-9.0% of mass 174
176	> 95% but < 101% of mass 174	> 95% but < 101% of mass 174	93.0-101.0% of mass 174
177	5-9% of mass 176	5-9% of mass 176	5.0-9.0% of mass 176

(1) *8260 criteria taken from CLP OLMO4.0 (January 1998)

Initial calibration (ICAL) is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector.

After the initial calibration standards are injected, a calibration curve is constructed using internal standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD of the calibration curve is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard (CCV) must be analyzed at the required interval as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Volatiles		
Method	**Initial Calibration Check Criteria**	**Continuing Calibration Check Criteria**
624	All targets <= 35% RSD, or alternatively, construct calibration curve	QC Check Sample (20 µg/L) meets limits specified in method –Table 5, Range for Q
8260	CCC <= 30% RSD	CCC <= 20% difference or drift from initial calibration
	SPCC (minimum RF) Chloromethane 1,1-Dichloroethane Bromoform Chlorobenzene 1,1,2,2-Tetrachloroethane	 0.10 0.10 >0.10 0.30 0.30 (0.10 for 25-mL purge) (1)
524.2	All targets <= 20% RSD, or alternatively, generate linear, 2nd or 3rd order calibration curve	All targets <= 30% difference from initial calibration, or alternatively, using analyst judgment, all targets must fall on the initial calibration curve

(1) The purging efficiency of 1,1,2,2-tetrachloroethane relative to the internal standard is such that the SPCC criteria cannot be met consistently for a 25mL purge. The response factor is generally in the 0.1 to 0.3 range. The alternate criteria is adopted from the EPA CLP Low Level Statement of Work, a protocol similar in scope and application to SW-846 Method 8260.

SW-846 Method 8260: After the CCC and SPCC are evaluated, all target compounds are evaluated for linearity. If the %RSD is less than or equal to 15%, the average response factor can be used for quantitation. If the %RSD exceeds 15%, the "grand mean" exception can be applied to the ICAL. Alternatively, a regression curve (linear, quadratic, etc.) may be used for quantitation if the correlation coefficient or coefficient of determination is greater than 0.99.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 15%.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 20%.

Semivolatile GC/MS (SM)
Semivolatile organic compounds (SVOC) are analyzed by four GC protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; 8000-series are primarily for RCRA testing; and the CLP protocols are followed for pesticides for hazardous waste site monitoring.

Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable EPA methods. Ion abundance acceptance criteria for SVOC tuning with DFTPP are given below. Mass calibration is performed as an integral part of tuning.

The tune check and calibration check must be performed in the following intervals:

625 - Daily
8270/CLP - every 12 hours.
525.2 - every 8 hours.

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)			
	Ion Abundance Criteria		
m/e	525.2	625	8270/OLMO4.0 (1)
51	10-80% of mass 442	30-60% of mass 198	30-80% of mass 198
68	Less than 2% of mass 69	Less than 2% of mass 69	Less than 2.0% of mass 69
69	(reference only)	(reference only)	Present
70	Less than 2% of mass 69	Less than 2% of mass 69	Less than 2.0% of mass 69
127	10-80% of mass 198	40-60% of mass 198	25-75% of mass 198
197	Less than 2% of mass 198	Less than 1% of mass 198	Less than 1% of mass 198
198	Greater than 50% of mass 442	Base peak, 100% relative abundance	Base peak, 100% relative abundance
199	5-9% of mass 198	5-9% of mass 198	5.0-9.0% of mass 198
275	10-60% of mass 442	10-30% of mass 198	10-30% of mass 198
365	Greater than 1% of mass 442	Greater than 1% of mass 198	Greater than 0.75% of mass 198
441	0-100% of mass 443	Present but less than mass 443	Present but less than mass 443
442	Base peak, 100% relative abundance	>40% of mass 198	40-110% of mass 198
443	15-24% of mass 442	17-23% of mass 442	15.0-24.0% of mass 442

(1) *8270 criteria taken from CLP OLMO4.0 (January 1998)

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector.

After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD of the calibration curve is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard must be analyzed daily as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Semivolatiles - GC/MS		
Method	**Initial Calibration Check Criteria**	**Continuing Calibration Check Criteria**
625	All targets <= 35% RSD, or alternatively, construct calibration curve	All targets <=20% difference from initial calibration
8270	CCC <= 30% RSD; SPCC >= 0.050	CCC <= 20% difference from initial calibration SPCC >= 0.050
525	All targets <= 30% RSD, or alternatively, generate linear, 2nd order, or 3rd order calibration curve.	All targets <= 30% difference from initial calibration, or alternatively, using analyst judgment, all analytes must fall on the initial calibration curve

SW-846 Method 8270: After the CCC and SPCC are evaluated, all target compounds are evaluated for linearity. If the %RSD is less than or equal to 15%, the average response factor can be used for quantitation. If the %RSD exceeds 15%, the "grand mean" exception can be applied to the ICAL. Alternatively, a regression curve (linear, quadratic, etc.) may be used for quantitation if the correlation coefficient or coefficient of determination is greater than 0.99.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 15%.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 20%.

9.4.5 High Performance Liquid Chromatography (LC)

Semivolatile organic compounds (SVOC) are analyzed by three LC protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; and 8000-series are primarily for RCRA testing. If internal standard calibration is used; relative retention time, as defined in the respective SOPs, will be used to determine the identification of the target compounds and bracketing by CCV will not be required unless specified in the method or QAPP. If external standard calibration is used, the absolute retention time window is calculated as three times the standard deviation obtained from a 72-hour sequence or default windows of 0.05 to 0.10 minutes are used for compounds where the calculated window is too restrictive or zero. Bracketing by CCV will be required for external standard calibrations if specified in the method, SOP, or QAPP.

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD of the calibration curve is less than or equal to the acceptance criteria, the average response factor can be used for quantitation. A midpoint calibration verification standard must be analyzed daily as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Calibration Check	500-series	600-series	8000-series
Initial calibration -minimum calibration standards	3 (as the calibration range is extended, the number of points must be increased)(2)	3	5
%RSD criteria(1)	<=20%	<=10%	<=20% (with exceptions noted below)
CCV criteria (%difference or %drift)	+/- 20%	+/-10%	+/-15% (with exceptions noted below)
Frequency of CCV	Every 8 hours	Daily	Every 12 ours

(1) Alternatively, a regression curve (linear, quadratic, etc.) may be constructed. If the correlation coefficient of the regression curve is greater than or equal to 0.99, the curve may be used for quantitation of samples.
(2) An alternate single point calibration can be performed if the standard response is within 20% of the sample response.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

9.4.6 Gas Flow Proportional Counter

Initial calibration is performed at instrument start up, following instrument maintenance or a change in quench gas. Counting voltage is set utilizing a sealed beta source by increasing the voltage through the detector until the beta plateau region is determined. The amount of cross talk into the alpha channel is then minimized and the beta count maximized utilizing the discriminator. Counting efficiencies are then established utilizing the (relatively) pure beta and alpha source standards. These standards are analyzed daily, and re-calibration is required if their activity count varies 5% or more.

Self-absorption curves are generated at a frequency of once every three months, and the results are plotted on a cumulative scatter plot curve. Values for self-absorption are then taken from the best-fit curve generated from this plot and applied to all sample result calculations. Duplicate laboratory control samples are analyzed with each batch of 20 samples to determine accuracy and precision. Additionally, a matrix spike and matrix spike duplicate or sample duplicate are analyzed with each batch of samples. Instrument background counts are also performed daily for use in all sample result calculations for that day.

9.4.7 Radon Flask Counter

Initial calibration is performed at instrument start up or following instrument maintenance. The alpha plateau region is initially determined utilizing a sealed source alpha emitter. Each radon flask is then evacuated, filled with helium at atmospheric pressure, and background counted. Their individual efficiencies are then determined utilizing a standard of known activity. The sealed source alpha emitter is counted daily, and re-calibration is required if its activity varies by 10% or more.

Duplicate laboratory control samples are analyzed with each batch of 20 samples to determine accuracy and precision. Additionally, a matrix spike and matrix spike duplicate or sample duplicate are analyzed with each batch of samples.

9.4.8 Liquid Scintillation Counter

Initial calibration of this instrument is performed at the factory. Following set up and prior to use, the calibration is verified using unquenched Carbon-14 Standard and background water. Quench curves are also factory installed.

9.5 Low Level Calibration Check

An additional optional continuing calibration check standard at or below the RL (usually lowest level standard) may be analyzed daily. This check standard is used to demonstrate that the RL can be achieved and may also be used in conjunction with the calibration check standard for reporting data. If the mid point CCV standard fails acceptance criteria and a standard at the reporting limit (RL) is analyzed within the same analytical clock, samples which have no target compounds detected above the RL may be reported as <RL, if

1) all targets in ht eRL standard are detected at the established retention time
2) the surrogate recovery, if applicable, is within acceptance limits
3) the internal standard area(s), if applicable, is within acceptance limits

9.6 Field Instrument Calibration

Calibration of field instrumentation (conductivity/salinity meters, pH meters, DO meters, and turbidimeters) is performed in the field prior to use, in accordance with the manufacturers and method specifications. All calibration data are documented in a bound field notebook.

9.7 Calibration Documentation

All calibration records including raw data, response factors, standard concentrations, curves, reduced data, and instrument settings or conditions are stored and archived as hard or electronic copy according to laboratory standard operating procedures. Current chromatograms, curves, and results transcribed onto forms are kept at the analysts' workstations and periodically archived into a data storage area. Initial and continuing calibrations are stored by date for ease of location. All standard ID numbers appear on graphs, plots, chromatograms, or curves for traceability purposes. SL SOP QC16: *Analytical Records Maintained by SL* describes the policies and procedures for the archival of raw data and associated QC data.

9.8 Thermometer Calibration and Temperature Checks

Equipment such as refrigerators, ovens, waterbaths, hot plates, and incubators are periodically checked with calibrated thermometers. Refrigerators and waterbaths are checked daily and the temperatures documented in a notebook. The temperature of microbiological incubators must be checked and recorded twice daily Sample storage refrigerators should be set to 4 C but must be less than 6C, but must not freeze aqueous samples. All thermometers are calibrated annually against an NIST-certified thermometer.

9.9 Balance Calibration

Electronic analytical balances are calibrated daily with internal mechanisms, if available. The calibration of the balance must be checked daily by the analysis of two Class S weight that bracket the approximate weight of material that is being determined. The balance must be checked weekly by the analysis of the weights in Table 9.5 that the lab routinely determines. The daily and weekly calibration checks must be documented in a bound logbook kept with the balance in accordance with SL SOP AN10.

TABLE 9.1
LABORATORY INSTRUMENTS AT EACH SAVANNAH LABORATORIES LOCATION

#	Instrument	Savannah	Tallahassee	Tampa	Mobile
6	ICP Units	3	1	1	1
4	Mercury Cold Vapor Units	1	1	1	1
12	Atomic Absorption Furnace/Flame	5	3	2	2
32	GC/MS	16	7	4	5
60	Gas Chromatographs	20	18	12	10
33	Flame Ionization Detectors(FID)	12	8	8	5
17	Photoionization Detectors(PID)	4	5	5	3
17	Hall Electrolytic Conductivity Detectors (HECD)	5	4	5	3
1	Thermal Conductivity Detectors(TCD)	1			
34	Electron Capture Detectors(EC)	12	10	6	6
6	Nitrogen-Phosphorous Detectors(NPD)	2	2		2
24	Purge and trap Devices for Liquids	12	8	6	3
6	Purge and trap Devices for Soils (5035)	3	1	1	1
3	TOC Analyzers	1	1		1
5	IR Spectrophotometers	2	1	1	1
9	UV-VIS Spectrophotometers	3	2	2	2
6	Nutrient Autoanalyzers	3	1	1	1
6	HPLC Units	2	4		
2	Ion Chromatograph	1	1		
1	Alpha-Beta Counter			1	
1	Scintillation Counter			1	
2	Radon flask Counter			2	
2	Scaler			2	

TABLE 9.1
LABORATORY INSTRUMENTS AT EACH SAVANNAH LABORATORIES LOCATION

#	Instrument	Savannah	Tallahassee	Tampa	Mobile
23	PH/ISE Meters	12	4	2	3
11	DO Meters	4	2	3	2
8	Turbidimeters	2	2	2	2
4	TOX/AOX Analyzers	3			1
9	Conductivity Meters	3	2	2	2
1	Bomb Calorimeter	1			
7	Analytical Balances	2	2	1	2
21	Top Loading Balances	7	5	4	5
4	Autoclaves	1	1	1	1
18	Waterbaths	4	7	3	4
8	Biological Incubators	2	2	2	2
7	BOD Incubators	3	1	1	2
26	Drying Ovens	7	9	4	6
11	Block Digestors	5	3	1	2
4	TCLP (Nonvolatile) Extractor Tumblers	SL Custom	SL Custom	SL Custom	SL Custom
4	TCLP (ZHE) Tumblers	2	1		1
11	Sonicators	4	3	2	3
13	Sample Concentrators	4	3	3	3
4	Gel Permeation Chromatographs (GPCs)	2	2		

TABLE 9.2

MAJOR FIELD INSTRUMENTS AT EACH SAVANNAH LABORATORIES LOCATION

#	Instrument	Tallahassee	Savannah	Mobile	Tampa Bay
3	pH/SC/DO/T Meters		1-Corning Checkmate 90		1-Corning Checkmate 90
9	pH/Temp Meters	2-Orion 230A	1-Orion SA-230	3-Orion 23A, 1-Orion 290A	1-Orion 23A
8	Conductivity/ Salinity Meters	1-YSI 33	1-YSI 33	2-YSI 33 1-Orion 120	1-YSI 33
7	DO Meters	1-YSI 51B	1-YSI 51B	1-YSI 50B 1-YSI 51B	2-YSI 50B
4	Turbidimeters	1-Hach 16800	1-DRT 15C	1-Hach 2100P	1-Hach 16800
8	Water Level Meters	1-Slope 51453	1-Fisher	4-Solinst	1-Rocktest Model CPR6
1	Residual Chlorine Colorimeter			1-Hach Pocket Colorimeter	

TABLE 9.3
STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
ICP	Baker/Spex	Stock 1,000 or 10,000 ppm solutions	Room temp	Working std prepped directly from stock	Room temp	Monthly or as needed
AA	Baker/Spex	Stock 1,000 ppm solutions	Room temp	Intermediate stds prepped from stocks. Working stds prepped from intermediates.	Room temp Room temp	Daily Daily
Autoanalyzer	Fisher Baker	Neat material	Room temp	Stock stds prepped from solids. Intermediate stds from stocks. Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed
Ion Chromatograph	Fisher Baker Mallinckrodt	Neat material	Room temp	Stock stds prepped from solids. Intermediate stds from stocks. Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed
UV-VIS Spectrophotometer	Fisher Baker EM	Neat Material	Room temp	Stock stds prepped from solids. Intermediate stds from stocks. Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed

TABLE 9.3
STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
IR Spectrophotometer	Fisher	Neat liquids	Room temp	Stock std prepped from neat liquid. Working stds from stock.	Refrigerator Refrigerator	Monthly Monthly
Turbidimeter	Hach	Standard 4000 ppm formazin solution	Refrigerator	Working stds prepped from stock.	Used immediately	As needed to check Gelex stds
Conductivity Meter	YSI or Fisher	Standard solution or neat KCl	Room temp	Used as is or prepare from neat.	Room temperature	As needed
TOC	Mallinckrodt	Neat KHP	Room temp	Stock std from solid Working std from stock.	Refrigerator Refrigerator	Monthly Monthly
pH Meter	Fisher	Calibration buffer solutions	Room temp	Used as is.	----	----
Ion Selective Electrode (ISE)	Baker	Neat material	Room temp	Stock std from source. Intermediate std from stock. Working std from intermediate.	Refrigerator Refrigerator Used immediately	Monthly Monthly or as needed As needed

TABLE 9.3
STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
TOX	Fisher	Neat material	Room temp	Std from source.	Room temp	Monthly
Bomb Calorimeter	Parr	Neat tablets	Room temp	Used as is.	----	----
GC and GC/MS (Volatiles)	Supelco, Ultra, Accustandard, ChemService, Baxter, Aldrich, Restek, NSI	Neat Solutions (50-5000 ppm)	Freezer	-Stock stds from neat sources. -Intermediate stds from stocks. -Working standards from intermediates and/or purchased solutions.	Freezer Freezer Freezer	-Annually or manufacturer expiration -Semi-annually; monthly for reactive compounds -Weekly
GC and GC/MS (Semivolatiles)	Supelco, Restek, ChemService, Crescent Chemical, Aldrich, Ultra, NSI	Neat Solutions (50-10000 ppm)	Refrigerator	-Stock stds from neat sources. -Intermediate stds from stocks. -Working standards from intermediates.	Refrigerator or freezer Refrigerator or freezer Refrigerator or freezer	Semi-annually or annually as required Semi-annually or annually as required Semiannually or as needed
HPLC	ChemService, Chemical, Supelco Accustandard Radian Absolute	Neat 1000 ppm 1000 ppm 1000 ppm 1000 ppm	Refrigerator	-Stock stds from neat sources. -Intermediate stds from stocks and/or purchased solutions. -Working standards from intermediates.	Refrigerator Refrigerator Refrigerator	Semi-annually Monthly Weekly

TABLE 9.3
STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
Gas Proportional Counter (alpha/beta)	EPA NIST	Sealed Source Stock Soln.	Room temp Metal case Room temp Foil-lined cabinet	Used as is. Working std, prepped from stock.	– Room temp Foil-lined cabinet	– As needed
Radon Flask Counter	EPA NIST	Sealed Source Stock Soln.	Room temp Foil-lined cabinet Room temp Foil-lined cabinet	Used as is. Working std, prepped from stock.	-- Room temp Foil-lined cabinet	– As needed

TABLE 9.4
STANDARDIZATION OF TITRATION SOLUTIONS

Analysis	Solution Requiring Standardization	Standard Identity	Standard Source	Frequency of Standardization
Acidity	Sodium Hydroxide (0.02 N)	KHP	Mallinckrodt	With each batch
Alkalinity	Sulfuric acid	Na_2CO_3	Mallinckrodt	With each batch (or purchased certified)
COD	Ferrous ammonium sulfate	$K_2Cr_2O_7$	Mallinckrodt	With each batch
Chloride	Silver nitrate	NaCl	Baker	With each batch (or purchased certified)
Sulfide	Sulfide working standard	$I_2/Na_2S_2O_3$	VWR/Baker	Weekly
TOC (Soil)	Ferrous sulfate	$K_2Cr_2O_7$	VWR/Baker	With each batch

TABLE 9.5	
BALANCE CALIBRATION CHECKS	
Analytical Balance	
Class S Weight	**Tolerance**
0.01 g	± 0.0002 g
0.1 g	± 0.0002 g
0.5 g	± 0.0004 g
1 g	± 0.0004 g
10 g	± 0.0005 g
50 g	± 0.0010 g
Top-Loading Balance	
Class S Weight	**Tolerance**
0.1 g	± 0.02 g
0.5 g	± 0.02 g
1 g	± 0.04 g
5 g	± 0.04 g
10 g	± 0.05 g
50 g	± 0.20 g
100 g	± 0.20 g
300 g	± 0.50 g

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10.0 PREVENTIVE MAINTENANCE

10.1 Maintenance Schedule

All Savannah Laboratories facilities are equipped with up-to-date computerized instrumentation. In order to gain maximum performance and minimize downtime, regular inspection, maintenance, cleaning, and servicing of all laboratory and field equipment is performed according to the manufacturers' recommendations. A maintenance log is kept for each piece of laboratory and field instrumentation, detailing all maintenance performed on the instrument. Routine repairs and maintenance are performed and documented by the analyst responsible for the particular instrument. Non-routine maintenance is signed and dated by the analyst or repair technician. Routine maintenance procedures for laboratory instrumentation are given in Table 10.1 and each SOP. The frequencies of routine maintenance procedures for Savannah Laboratories' field instrumentation are given in Table 10.2. The service intervals listed in Tables 10.1 and 10.2 are as follows: D=daily; W=weekly; M=monthly; Q=quarterly; SA=semi-annually; A=annually; AN=as needed.

Maintenance contracts are carried for most instrumentation, and close contact is maintained with service personnel to provide optimum instrument functioning.

An extensive spare parts inventory is maintained for routine repairs at the facilities, consisting of GC detectors, AA lamps, fuses, printer heads, flow cells, tubing, certain circuit boards and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be exchanged among the labs.

10.2 Contingency Plan

In general, each facility has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation within each facility, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Savannah Laboratories location (where identical SOPs, QA procedures and instruments are utilized). When shipping samples to another facility, interdivisional chain-of-custody procedures are followed as given in Section 7.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
ICAP								
Pump Tubing	X							Change.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Quartz Torch							X	Clean and realign.
D-Shaped Mirrors			X					Inspect monthly, clean or replace as needed.
SMITH-HIEFTJE FURNACE AA SPECTROPHOTOMETER								
Sapphire Window	X							Remove and clean.
Flow Rate	X							Check.
Graphite Tube							X	Replace.
Quartz Windows	X							Clean.
Contact Rings and Plates	X							Clean daily, replace if worn.
Filters			X					Inspect monthly, clean or replace as needed.
ZEEMAN FURNACE AA SPECTROPHOTOMETER								
Sampler syringe	X							Check for air daily, flush syringe as needed.
Graphite Tubes	X							Inspect daily, replace as needed.
Graphite Electrodes				X				Inspect quarterly, replace if worn.
Quartz Windows	X							Remove and clean.
LEEMAN PS200 MERCURY ANALYZER AND AUTOSAMPLER								
Pump Tubing	X							Inspect daily, replace as needed.
Standard Cup	X							Inspect daily, replace as needed.
Drying Tube	X							Repack daily at a minimum.
Mixing Coil		X						Inspect weekly, clean or replace as needed.
Sample Probe			X					Inspect monthly, clean or replace as needed.
Mercury Lamp							X	Clean or replace.
LEEMAN AP200 PREP STATION								
Autosampler		X						Clean and oil rails.
Tubing					X			Inspect semi-annually, replace as needed.
Bottle Caps				X				Inspect quarterly, replace as needed.
Dispenser				X				Inspect semi-annually, replace as needed.
Water Bath			X					Clean.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
CEM MDS 2100 MICROWAVE								
Pressure Control System	X							Flush.
Cavity and exhaust		X						Clean.
Door			X					Inspect seals and locks.
CONTINUUM FURNACE AA SPECTROPHOTOMETER								
Quartz Windows	X							Remove and clean.
Graphite Tubes	X							Inspect daily, replace as needed.
Contact Rings	X							Clean daily and replace if worn.
Filters			X					Inspect monthly, clean or replace as needed.
D2 Arc Lamp							X	Adjust or replace.
TURBIDIMETER				X				Standardize against formazin.
CONDUCTIVITY METER							X	Replatinize cell when 1 umho/cm range exceeds 90-100%, and when erratic readings cannot be corrected.
pH METER							X	Clean or replace probe.
TOX ANALYZER								
Pyrolysis Tube							X	Clean or replace.
Electrodes							X	Clean.
Electrolytes	X							Replace.
ION CHROMATOGRAPH								
Separator Column				X				Clean.
Guard Column				X				Clean.
Pump Pistons						X		Inspect.
Conductivity Cell						X		Clean.
AUTOANALYZER (TRAACS/LACHAT)								
Pump Platen							X	Replace.
Pump Tubes							X	Replace.
Flow Cell							X	Inspect and clean.
BLOCK DIGESTOR						X		Check calibration.
UV/VIS SPECTROPHOTOMETER					X			Check for wavelength verification.
ION SELECTIVE ELECTRODE							X	Polish electrode.
BOMB CALORIMETER							X	Replace seals.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
AUTOCLAVE								
Pressure Verification	X							Check and document; replace gauge or seals as needed.
Drain Autoclave	X							Visually inspect for leaks and signs of degradation.
Temperature Verification		X						Check with autoclave thermometer; document; replace thermometer as needed.
Cleaning		X						Wash with soapy water; visually inspect for leaks and degradation.
Times				X				Check with stop-watch; replace as needed.
Seals							X	Visually inspect and replace as needed.
TOC ANALYZER								
Catalyst							X	Replace.
Meters (Internal)			X					Check against internal flow meter.
Detector Windows		X						Check and clean.
Humidifiers							X	Fill levels.
Syringe			X					Perform zero point calibration.
GAS CHROMATOGRAPH - SEMIVOLATILES								
Autosampler System							X	Syringe and tubing cleaned. Needles and tubing replace.
Septa	X							Replace as needed.
Column/Injector							X	Change sleeve and cut front of guard column. Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - MASS SPEC SEMIVOLATILES								
Column/Injector							X	Change sleeve and cut front of column.
Septum	X							Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
Splitless Disc							X	Replace.
Autosampler							X	Syringe and tubing cleaned. Needles and tubing replaced.
Rough Pump							X	Oil changed by HP service.
Mass Spectrometer							X	Clean.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Tape Head							X	Clean.
Tape Drive							X	Clean.
GAS CHROMATOGRAPH - VOLATILES								
Column							X	Replace.
Septum							X	Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - MASS SPEC VOLATILES								
Column							X	Replace.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
Rough Pump							X	Oil change by HP service.
Gas Manifold	X							Inspect daily, change when pressure reads <500 psi.
Tape Head							X	Clean.
Tape Drive							X	Clean.
GAS CHROMATOGRAPH - SCREENING FOR VOLATILES								
Autosampler							X	Syringe and tubing cleaned. Needles and tubing replaced.
Septa	X							Replace as needed.
Column							X	Replace.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - DAI								
Autosampler							X	Syringe and tubing cleaned. Needles and tubing replaced.
Column							X	Replace.
Septa	X							Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - MASS SPEC AIR ANALYSIS								
TDC Detector								
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Filament	X							Checked for proper temperature when analysis required.
Injector Loop	X	X						Change daily for analysis of atmospheric gases. Change weekly for analysis of non-atmospheric gases.
Column							X	Replace.
Mass Spec								
Cryo-column	X							Inspect for breaks.
Septa		X						Replace as needed.
Column							X	Replace.
Rough Pump							X	Oil change by HP service representative.
Mass Spectrometer							X	Clean.
PURGE AND TRAP								
Sorbent Trap					X			Inspect semi-annually. Adjust as needed.
Purge Flow								
HPLC SYSTEMS								
Pumps	X							Pressure check daily, change guard column as needed. Visual leak check daily.
Pump Seals				X				Inspect seals quarterly, replace as needed.
Column	X							Pressure check. Visual leak check.
Detector Fittings	X							Visual leak check.
Detector Optics				X				Remove and inspect filter quarterly, clean or replace as needed.
Autosampler	X							Check seal pack for leaks.
ZYMARK EXTRACT CONCENTRATOR								
Bath		X						Change water and scrub bath; dust outside.
Temperature Verification		X						Verify bath temperature and adjust.
Sensor Diagnostic Test		X						Check each position and adjust.
TENNELEC LB5100								
Sample Change				X				Inspect moving parts quarterly, lubricate as needed.
Detector	X							Inspect for proper operation and response. Serviced by manufacturer only.
Detector gas			X					Inspect monthly, change tank when pressure reads <500 psi. Allow new tanks to dissipate radon for two weeks before use.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
Flow Meter	X							Inspect for proper operation.
BECKMAN LS6500 Liquid Scintillation Counter		X						Inspect for proper operation prior to use. Serviced by manufacturer only.
LUDLUM MEASUREMENTS 2000	X							Inspect for proper operation prior to use. Serviced by manufacturer only.
LUDLUM MEASUREMENTS 182	X							Inspect push rod for high voltage engagement. Inspect instrument noise level without flask.
TCLP EQUIPMENT								
Volatile Rotator			X					Check rotation.
Semivolatiles/Metals Rotator		X						Check rotation.

TABLE 10.2

FIELD EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval					SERVICE LEVEL
	D	W	M	Q	A	
TURBIDIMETER HACH 16800/DRT-15C	X					Inspect daily prior to sampling and replace cell as needed.
CONDUCTANCE METER-YSI 33	X					Inspect daily, replatinize cell as needed.
pH METER	X					Inspect daily prior to sampling, add filling solution as needed.
CORNING CHECKMATE 90 pH/SC/DO/T° METER	X					Inspect probe, membrane, battery daily prior to sampling. Change parts as needed.
YSI MODEL 50B/51B DISSOLVED OXYGEN METER	X					Inspect probe daily prior to sampling, change as needed.
WATER LEVEL INDICATOR	X					Inspect probe and meter prior to use at every well.

11.0 QUALITY CONTROL CHECKS AND ROUTINES TO ASSESS PRECISION AND ACCURACY AND CALCULATIONS OF METHOD DETECTION LIMITS

The key to a successful QA/QC program is strict adherence to the program during all phases of the project, including pre-sampling discussions; sample collection, preservation, storage and analysis; and validation and reporting of results. Field and laboratory quality control checks, which are part of each sampling trip and laboratory analysis, meet or exceed all agency requirements. Without the proper quality control procedures, analyst and method performance cannot be measured.

If project-specific quality assurance plan (QAPP) QC requirements are more stringent than the general procedures given below, QAPP requirements are followed.

11.1 Field QC Checks

Savannah Laboratories recommends to their clients that proper control procedures meet or exceed the appropriate regulatory agency field QC requirements.

Blanks, which are collected in the field, are an important link in the quality control data chain for a set of samples. The analytical data derived from these blanks are necessary to assess field-sampling operations. These blanks are used to verify that sample containers, preserving reagents and equipment are contaminant-free. Blanks are also used as a check for potential on-site environmental contamination, to evaluate personnel expertise in sample collection, and to reveal problems that may occur in sample storage and transport.

The field quality control blanks should not be isolated from actual samples. They must be considered as samples and treated identically (preserved with the same reagents, stored and transported in the same containers as the samples, etc.).

The types and frequency of blanks should be included in all quality assurance plans. In cases where data quality objectives dictate more stringent controls, additional field quality control blanks may be required. The following protocol outlines the minimum field blank requirements necessary to assure the validity and integrity of any sampling episode.

Field QC check samples will be analyzed according to the client's instructions and invoiced as samples. Since field QC check samples are usually liquids, they are prepared and analyzed by liquid procedures and reported as liquids. However for batching purposes, unless requested by clients or required by a project specific QA plan, lab QC deliverables are not provided for field QC check samples. Liquid QC samples are batched with soil samples for methods where preparation procedures are the same for both matrices (i.e., volatiles, cyanide, etc.).

11.1.1 Trip Blanks

PURPOSE: The trip blank is to be used when sampling for volatile organics. The purpose is to determine if contamination has occurred as a result of improper sample container cleaning, contaminated blank source water, sample contamination during storage and transportation due to exposure to volatile organics (e.g., gasoline fumes) and other environmental conditions during the sampling event.

PREPARATION: Trip blanks are prepared prior to the sampling event either by the laboratory providing sample containers, or by field team personnel who are responsible for the initial preparation of sample containers and field equipment. The water must be free of volatile organic contaminants. Any appropriate preservatives must be added at the time that the blanks are prepared. The sample containers are sealed, labeled appropriately, and transported to the field in the same sampling kits as the sample vials. These blanks are not to be opened in the field. They are to be transferred to the sample container designated for volatile sample storage, and transported with the samples to the laboratory.

FREQUENCY: One trip blank for each volatile organic analysis (601, 602, 624, 8021, etc.) should be provided per cooler used for storing and transporting volatile sample vials. If a laboratory requires submission of multiple vials for a method, the same number of vials must be submitted for the trip blank.

11.1.2 Field Blanks

PURPOSE: Field blanks are used to evaluate the effects of on-site environmental contaminants, the purity of reagents used as preservatives or additives, and the general sample collection techniques. Field blanks are recommended for all parameters but are not mandatory.

PREPARATION: Field blanks are prepared on-site by filling the sample container(s) with analyte-free water, adding preservatives, sealing the containers and completing the appropriate documentation. The field blanks must be handled in the same manner as the sample group for which it was intended (i.e., blanks must be stored and transported with the sample group).

FREQUENCY: One field blank per parameter group per day or at a frequency of 5% of the samples in the parameter group per day, whichever is greater.

11.1.3 Equipment Blanks

PURPOSE: Equipment blanks are recommended if sampling equipment is field-cleaned. These blanks are used to determine the effectiveness of field cleaning procedures as well as to reveal those sources of contamination that may be found in a trip blank. Equipment blanks must be collected and analyzed for all parameter groups and matrices.

PROCEDURE: The final rinse water (analyte-free) shall be rinsed on or through the sampling equipment, whether pre-cleaned or field cleaned, collected, and placed in appropriate preserved containers. These blanks must be stored and transported with the samples.

FREQUENCY: When less than five samples of a similar matrix are taken, one equipment blank prepared on-site for pre-cleaned or field-cleaned equipment should be collected and analyzed for each parameter. When five to ten samples of a similar matrix are taken, one equipment blank should be collected on field-cleaned equipment or one on-site blank should be collected in pre-cleaned equipment if no equipment is cleaned in the field.

For sampling events involving ten or more samples, a minimum of one blank should be taken on pre-cleaned equipment or at the rate of 5% (whichever is greater) of the samples in each analyte group for all matrices. One blank should be taken on field-cleaned equipment or at the rate of 5% (whichever is greater) of the samples in each analyte group for all matrices.

11.1.4 Field Duplicates

Field duplicates are taken, analyzed, reported and invoiced as required. A minimum of one duplicate for 10% of samples should be taken for all parameter groups and matrices to be collected and analyzed.

11.1.5 Field QC Summary

The recommended frequency of field blanks and duplicates is summarized below:

No. Samples	Pre-cleaned Equipment Blanks	Field-Cleaned Equipment Blanks	Trip Blank (VOCs)	Duplicates
10+	Minimum of one, then 5%	Minimum of one, then 5%	One per cooler	Minimum of one, then 10%
5-9	One*	One*	One per cooler	One
< 5	One*	One*	One per cooler	Not required

* Note: For nine or fewer samples, one equipment blank is recommended from either pre-cleaned or field-cleaned equipment.

If any equipment is cleaned in the field, the blank should be taken from the field-cleaned equipment.

11.2 Laboratory QC Checks

The laboratories employ control samples to assess the validity of the analytical results. Determination of the validity of sample results is based on the acceptance criteria being met by the control samples. The acceptance criteria for each type of control sample are defined in the appropriate SOP. These acceptance criteria are per method requirements or calculated annually from historical data.

Matrix spike results will be utilized for laboratory control when specified by the method. If matrix spikes are out of control, or control is based on laboratory control standards (LCS), then LCS results and method control criteria will ultimately be used to accept or reject the analytical batch. Clients are requested to provide sufficient sample for matrix spikes and are invoiced for matrix spikes.

For CLP protocols or other cases (i.e., when mandated by client or project specific QAPP) where "sample specific" (non-batch) QC is required, matrix spike/duplicate analysis will be conducted on replicate samples provided by the client. In all other cases, matrix spikes will be on a batch-specific basis (not client-, project- or sample-specific basis).

When possible, aliquots for matrix spikes are taken from the same container as the field sample. In some cases with liquid samples, this is not possible, i.e., semivolatile extractables, oil and grease, TPH, etc.

The control samples are analyzed in the same manner as the field samples. QC check samples include the following and are analyzed on an analytical batch frequency unless otherwise stated.

- Quality control check samples are analyzed in duplicate semiannually. These samples are analyzed as blind samples (e.g., WP and WS studies). See section 14.3.1.

- Quality control check standards are analyzed at a frequency equivalent to 5% of the samples in the analytical batch (or at a minimum of one in every 20 samples) in order to verify the analysis.

- Continuing calibration standards are analyzed at a frequency equivalent to 5% of the samples in the analytical batch (or at minimum of one in every 20 samples). Alternatively, the Quality Control check standard may be used to satisfy this requirement. At least one of the checks is a standard at a concentration of 1 - 2 times the laboratory practical quantitation limit (PQL) or reporting limit (RL).

An analytical batch is defined as a group of field samples which are processed as a unit. If the number of field samples in the group is greater than 20, each group of 20 samples or less is handled as a separate batch.

Other QC check samples are analyzed for performance evaluations or as part of internal or external audits as given in Section 14.

If QAPP or agency QC requirements are more stringent than the general procedures given below, QAPP or agency QC requirements are followed.

11.2.1 Organics

Method Blanks: A method blank will be analyzed for each batch of samples.

Lab Control Standards: A blank spike or lab control standard (LCS) will be processed and analyzed (per method requirement) with each batch of samples (except for CLP protocols and other methods which do not require an LCS). For drinking water samples, analyte spike concentrations will be at or near reporting limits as specified for lab-fortified blanks in the 500 series methods. A lab control standard duplicate (LCSD) will be prepared and analyzed if sufficient sample is not supplied for the MS/MSD or duplicate.

Surrogates: Appropriate surrogate(s) (see Tables 5.1 and 5.2) will be added to all samples, standards and blanks.

Matrix Spikes: Matrix spikes will be analyzed at a frequency of 5% of samples. If a method does not specify matrix-spiking compounds, the SW-846 or CLP matrix spiking compounds will be used. Appropriate matrix spikes will be used for other chromatographic methods in which matrix spikes are not defined. Matrix spikes containing all method-specified compounds should be analyzed monthly to generate accuracy and precision limits.

Matrix Spike Duplicates/Sample Duplicates: Duplicate samples or matrix spike duplicates will be analyzed at a frequency of 5% of samples. In cases where duplicate matrix spikes are used, precision data are obtained on only the matrix spiking compounds.

NOTE: Unless requested by the client, matrix spikes are not routinely performed on TCLP, SPLP, EPTOX., or waste dilutions.

Full List Spikes: For projects that require spiking a laboratory control sample (LCS) or matrix spike (MS) with all target compounds, re-extraction and/or re-analysis of the samples in the batch will not be performed if :

1) The recoveries of no more than one (1) compound when 5 to 10 compounds are spiked, two (2) compounds when 11 to 20 compounds are spiked, three (3) compounds when 21-30 compounds are spiked, or five (5) compounds when more than 30 compounds are spiked are determined to be outside the control limits, and recoveries for all spiked compounds are positive. When <5 compounds are spiked, all compounds should be within control limits.

2) The recovery of a spike exceeds the upper control limit (UCL) and the compound is not detected in any sample in the analytical batch.

As indicated in Methods 8260 and 8270, the following compounds have erratic recoveries under the routine conditions of the preparation and analytical procedures and will not be evaluated for corrective action nor included in the count (1) above if included in the LCS or MS:

VOC: Acrolein
Benzyl chloride
Carbon disulfide
2-chloroethyl vinyl ether
Pentachloroethane

SVOC: Aniline
Benzidine
Benzoic acid
Hexachlorocyclopentadiene
Hexachlorophene
Kepone
Alpha, alpha-Dimethylphenethylamine
Methapyrilene
4,4-Methylbis(2-chloroaniline)
p-Phenylenediamine

The above guidance is to used as the default for evaluation of full target spikes in organic analyses unless other corrective actions are defined in a project-specific quality assurance plan or in an SL pre-project plan.

11.2.2 Inorganic and General Chemistry

Calibration Blanks: Calibration blanks are non-digested blanks which are analyzed at a frequency of 10% of samples.

Method Blanks: Method blanks should be processed and analyzed with each batch of samples of the same matrix.

Lab Control Standards: A blank spike or lab control standard will be processed and analyzed with each batch of samples (except for CLP protocols and other methods which do not require an LCS). A lab control standard duplicate (LCSD) will be prepared and analyzed if sufficient sample is not supplied for the MS/MSD or duplicate.

Matrix Spikes: Matrix spikes will be analyzed at a frequency of 5% of samples.

Matrix Spike Duplicates/Sample Duplicates: Duplicate samples or duplicate matrix spikes will be analyzed at a frequency of 5% of samples.

NOTE: Unless requested by the client, matrix spikes are not routinely performed on TCLP, SPLP, EPTOX., or waste dilutions.

11.2.3 Microbiology

Quality control checks are routinely performed for all microbiological analyses. Strict requirements for the lab-generated deionized water must be met before it can be used in any testing. Each monitored parameter, its monitoring frequency, and its acceptance limits is as follows: residual chlorine, monthly, < 1.0 mg/L; trace metals (total Cd, Cr, Cu, Ni, Pb, Zn), annually, < 1.0 mg/L, individual metals < 0.05 mg/L; conductivity, daily < 2.0 umho/cm; heterotrophic plate count, monthly, < 500 CFU/mL; inhibitory residue, annually or for each new lot of detergent, less than 15% difference between groups; suitability, annually, ratio between 0.8 and 3.0.

Other laboratory QC practices are utilized to provide accurate microbiological results. Positive and negative microbiological controls are run with each new lot of medium. Autoclave tape is used to ensure proper sterilization of sample containers, media, etc. Incubators are maintained at 35 ± 0.5° C and water baths at 44.5 ± 0.2° C. Thermometers used for these monitoring purposes are calibrated annually against an NIST-certified thermometer. Other equipment, such as the dissecting microscope and colony counter, is maintained in clean operating condition at all times.

Microbiological samples are analyzed in duplicate at a rate of 10% of positive samples. A positive control sample is analyzed with each batch of coliform samples. A negative control is analyzed at least monthly. Additionally, all drinking water samples positive for total coliform must be confirmed. For environmental samples, 10% of samples positive for total coliform must be confirmed. A completed test for MPN analysis must be performed on 10% of all confirmed samples or at least quarterly.

Blanks are routinely analyzed with microbiological samples. For membrane filter analyses, sterile dilution water blank is run initially, after every 10 samples, and at the end of each analytical run. For MPN analysis, sterile dilution water is added to a lauryl tryptose broth tube for a blank for each analytical run.

11.2.4 Radiochemistry

Background Count: Background counts are obtained at a frequency of once per day for gross alpha, gross beta and radium 228; and determined for each flask prior to sample introduction for radium 226.

Method Blanks: Method blanks are analyzed at a frequency of 5% of samples of the same matrix.

Lab Control Standards: Lab control standards are analyzed with each batch of 20 samples of the same matrix.

Matrix Spike/Matrix Spike Duplicate or Sample Duplicates: These are analyzed with each batch of 20 samples of the same matrix.

11.3 Routine Methods Used to Assess Precision and Accuracy

A system for assessing precision and accuracy through tabulation (manual or electronic) is initiated for each parameter upon method validation. Control calculations are based on procedures in *The Handbook for Analytical Quality Control in Water and Wastewater Laboratories* (EPA, 1979) and contain both "warning limits" (± 2 standard deviations) control charts and "control limits" (± 3 standard deviations). Control limits are updated annually for all parameters. A minimum of ten data points is used to update these limits. Formulas used for calculations of precision and accuracy are provided in Section 5.0.

11.4 Method Detection Limits and Reporting Limits

Method detection limits (MDLs) are determined annually in accordance with the procedures in SW-846 and Appendix B of 40 CFR Part 136. This procedure includes analyzing seven or more (40 CFR Part 136) or a minimum of three (SW-846 Chapter One) prepared spikes or standards in reagent water at levels 3-5 times the estimated detection limit. The standard deviation of the replicate measurements is calculated, and the MDL is computed by multiplying by the appropriate Student's t value (n-1 degrees of freedom) for the appropriate 99% confidence level (for seven replicates, t = 3.14).

The MDL calculated by the procedure described above is defined as the minimum concentration of a substance that can be measured in reagent water and reported with a given confidence that the analyte concentration is greater than zero. SL makes no claim that the MDLs determined by this statistical procedure are obtainable in environmental samples.

For other protocols (i.e., Contract Laboratory), other procedures are used to estimate detection limits.

Since MDLs are based on the analyses of standards in reagent water, they may not be useful in reporting data for environmental samples; therefore, practical quantitation limits (PQL) or reporting limits (RL) are typically used for reporting a non-detected parameter. Reporting limits are defined as the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions and are determined to be the lowest concentration standard or the sample equivalent of the lowest concentration standard in the initial calibration.

The method detection limits and reporting limits are determined annually by the Corporate QA Manager in conjunction with Corporate Management and the division laboratory directors, laboratory managers, and QA Officers, from the data submitted by the four Savannah Laboratories' divisions.

Reporting limits for radiological analyses are from recommended values in EPA 40 CFR Chapter 1 (7-1-93 Edition) Section 141.

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12.0 DATA REDUCTION, REVIEW, AND REPORTING

12.1 Introduction

In order to provide the highest quality data possible, an extensive system for sample custody, data reduction, review, and reporting has been implemented.

12.2 Sample Custody

Upon receipt of the samples, the custody forms are checked against the sample identifications listed on the containers by the custody technicians, and a unique SL log number is assigned to each sample group. Any discrepancies are noted, including cooler temperatures, broken bottles and/or misidentified samples. Clients should be immediately notified if discrepancies exist.

After receipt, the samples are delivered to the appropriate laboratory sections where the samples are checked for proper preservation and this information is recorded in bound notebooks when applicable. When necessary, the samples are then stored in refrigerators that are monitored for temperature.

12.3 Organization and Initiation of Sample Analyses

The key to Savannah Laboratories' sample flow, analysis, data/QA review, archiving, and reporting system is the single LIMS network which controls the day to day production of the laboratories. This system, which is summarized in Figure 12.1, provides project managers, QA personnel, and all analysts immediate information on the status of any sample in all five facilities. This m schedules and prioritizes all work, provides a mechanism for sample tracking, review of reportables and QC data, generation eports and invoices, and archiving of all reports and associated QC data. The policies and procedures for the LIMS and other nputer systems are described in the current revision of the Savannah Laboratories' *Software Quality Assurance Plan.*

Upon receipt of custody forms, the project manager instructs data management personnel to log the sample analysis request and identification into the LIMS. The LIMS is based on an ADDS Mentor 7000 computer (NCR) which links the laboratories via telephone multiplex. This enables any project manager, section manager, QA manager, laboratory director, or analyst with authority to access and check the status of all projects.

If special handling or data packaging is required, the QA department and the laboratory receive copies of the custody forms and computer acknowledgments or a preproject plan. A sample delivery group (SDG) sheet is established and distributed to all affected departments including the various laboratory analysts, project managers, and section managers.

After the sample analysis request is logged into the LIMS and approved, the LIMS generates worksheets which are printed and distributed.

12.4 Sample Analysis and Data Reduction

Through the use of the worksheets and/or SDG sheets, the samples are prepared following the procedures given in each of EPA's approved methods. The preparation information is recorded in signed notebooks throughout the laboratory.

12.4.1 Data Reduction

Most sample concentration results are read directly from instrumentation without further reduction or calculations. Dilution factors are applied upon the dilution of samples having concentrations above the calibration range.

In many cases, these are input into the instrument computer and correct results are calculated automatically. In other cases, a manual calculation may be made. Soil/solid waste concentration results for all laboratory sections are calculated on a dry weight basis, prior to reporting, by dividing the instrument result by the fractional dry weight.

Other than the cases discussed above, data obtained by the following method/instrument are directly reportable: GC, GC/MS, metals, general chemistry automated colorimetry, TOC, DO, turbidity, and pH.

Data from methods requiring reduction prior to reporting include titrimetric methods, BOD, COD, conductivity, manual UV/VIS/IR, residue, TOX, and radiochemical parameters.

Table 12.1 gives equations used in computer-controlled instrumentation for data reduction as well as equations used for the manual calculation of reportable concentration results.

The laboratory raw data containing the instrument-generated reports, manually calculated results, and all supporting preparation, calibration, and analytical data are retained at the individual work stations until reports are issued unless additional handling or data packaging is required.

All pH and conductivity meters should be temperature compensated. Cell constants for field conductivity meters are determined by laboratory personnel annually as given in Section 9.4.2. Field conductivity is calculated as given in Table 12.1. All other field data are read directly from instrumentation.

Bound field notebooks are used for documentation of required data reduction. Calculations are recorded in waterproof ink.

12.4.2 Chromatographic and Data File Identification

Chromatograms and data files are given a unique alphanumeric identification by the chemists initiating the analyses in each section where appropriate. These file identification numbers reflect either the date the sequence was initiated (GC sections), the order in which the samples were analyzed (GC/MS sections), and/or the sample identification and log numbers given by the client and listed on the LIMS.

12.5 Data Transfer and Review

12.5.1 Data Transfer to LIMS

The analytical results are entered on the department worksheets after review or by direct electronic transfer from the instrument data system. After the data are entered into the LIMS, they are checked against the information entered into the LIMS for transfer errors and anomalies.

FIGURE 12.1 - FLOW CHART OF SL COMPUTERIZED LIMS

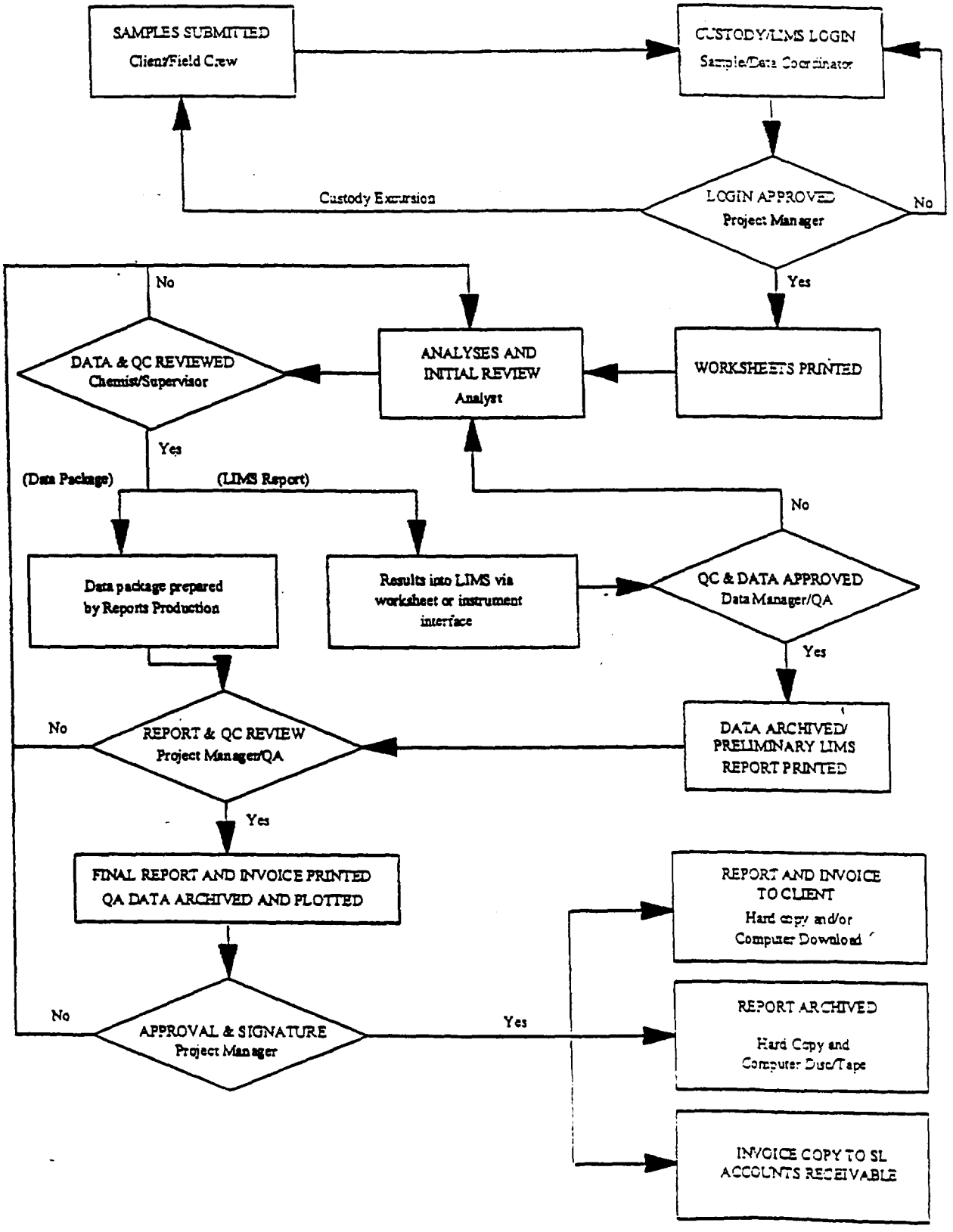

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporting Units	
N/A Extractables by GC/MS [Internal Standard Method (625 and 8270)]	Liquid	Solid
	ug/L (or mg/L)	ug/Kg (or mg/Kg)

$$RRF_s = \frac{A_S}{A_{IS}} \times \frac{C_{IS}}{C_S}$$

RRFs = relative response factor of standard
A_s = area of standard (area counts)
A_{is} = area of internal standard (area counts)
C_{is} = concentration of internal standard (ug/ml)
C_s = concentration of standard (ug/ml)

$$Water\ conc\ (ug/L) = \frac{A_S}{A_{IS}} \times \frac{C_{IS}}{RRF_S} \times \frac{V_f}{V_i} \times DF$$

V_f = final extract volume (ml)
V_i = initial sample volume extracted (L)
DF = dilution factor

$$Soil\ conc.\ (ug/kg) = \frac{A_S}{A_{IS}} \times \frac{C_{IS}}{RRF_S} \times \frac{V_f}{W_{spi}} \times \frac{1}{(\%solids \times 0.01)} \times DF$$

W_{spi} = weight of sample extracted (Kg)

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporting Units	
VOCs by GC/MS and GC	Liquid	Solid
	ug/L (or mg/L)	ug/Kg (or mg/Kg)

$$Relative\ Response\ Factor\ (RRF_S) = \frac{A_S}{A_{IS}} \times \frac{W_{IS}}{W_S}$$

A_s = area of standard (area counts)
A_{is} = area of internal standard (area counts
W_{is} = weight of internal standard (ug)
W_s = weight of standard (ug)

$$Water\ conc.\ (ug/L) = \frac{A_S}{A_{IS}} \times \frac{W_{IS}}{RRF_S} \times \frac{DF}{V_{spl}}$$

DF = dilution factor
V_{spl} = sample volume purged (L)

$$Soil\ conc.\ (ug/kg\text{-}dw) = \frac{A_S}{A_{IS}} \times \frac{W_{IS}}{RRF_S} \times \frac{DF}{W_{spl}} \times \frac{1}{(\%solids \times 0.01)}$$

W_{spl} = Weight of sample purged (Kg)

Pesticides/PCBs and Other GC Procedures	Liquid	Solid
	ug/L (or mg/L)	ug/Kg (or mg/Kg)

$$Response\ Factor\ (RF_s) = \frac{ug\ of\ standard}{peak\ area}$$

$$Water\ conc.,\ (ug/L) = RF_s \times peak\ area \times \frac{V_f}{V_i} \times \frac{DF}{V_j}$$

V_i = initial sample volume (L)
V_f = final extract volume (ml)
V_j = injection volume (ml)
DF = dilution factor

$$Sediment\ conc.,\ (ug/kg\text{-}dw) = RF \times peak\ area \times \frac{V_f}{(W_{spl})(\%solids \times 0.01)(V_j)}$$

W_{spl} = weight of sample extracted (Kg)

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporting Units	
Metals	**Liquid**	**Solid**
$$Water\ conc.\ (mg/L) = \frac{y - b}{m} \times \frac{V_f}{V_i} \times DF$$	ug/L (or mg/L) mg/L (or ug/L)	ug/Kg (or mg/Kg) mg/Kg (or ug/Kg)
y = $absorbance_{spl}$ b = y intercept from calibration curve (absorbance) m = slope from calibration curve (absorbance/(mg/ml) V_f = volum of digest (ml) V_i = volume of sample (L) DF = dilution factor		
$$Soil\ conc.\ (mg/kg\text{-}dw) = \frac{y - b}{m} \times \frac{V_f}{W_{spl}} \times \frac{1}{(\%solids \times 0.01)} \times DF$$		
W_{spl} = weight of sample (Kg)		
UV/VIS and IR Procedures	**Liquid**	**Solid**
$$Water\ conc.\ (mg/L) = \frac{y - b}{m} \times \frac{V_f}{V_i} \times DF$$	mg/L	mg/Kg
y = $absorbance_{spl}$ b = y intercept from calibration curve (absorbance) m = slope from calibration curve (absorbance/(mg/ml) V_f = final digest volume (mL) V_i = volume of sample (L) DF = dilution factor		
$$Soil\ conc.\ (mg/kg\text{-}dw) = \frac{y - b}{m} \times \frac{V_f}{W_{spl}} \times \frac{1}{(\%solids \times 0.01)} \times DF$$		
W_{spl} = weight of sample (Kg)		

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporting Units	
General Titrimetric Procedures	Liquid	
Analyte, mg/L = $\frac{N_{titrant} \times \text{Titer}}{\text{Vol. of sample titrated}}$ x eq. wt. x 1000	mg/L	
BOD	Liquid	
BOD, mg/L = $\frac{(\text{Int. DO} - \text{Final DO}) - \text{Seed Correction Factor}}{\text{Vol. fraction of sample}}$	mg/L	
C	Liquid	
COD, mg/L = $\frac{(\text{Blk titer} - \text{sample titer}) \times N_{FAS} \times 8000}{\text{Vol. of sample, mL}}$	mg/L	
Conductivity	Liquid	
Cell constant = $\frac{1000}{\text{Observed conductivity of 1000 - µS/cm std.}}$	µS/cm	
Residue	Liquid	
Residue, mg/L = $\frac{\text{Total wt.} - \text{Wt. of dish or filter}}{\text{Vol. of sample, L}}$	mg/L	
TOX	Liquid	Solid
TOX, µg/L = $(C_1 + C_2 - 2C_3) \times \frac{1000\ \text{mL}}{\text{Vol. of sample}}$ C_1 = instrument reading of 1 column C_2 = instrument reading of 2 column C_3 = instrument reading of blank column TOX, mg/kg = $\frac{\text{instrument reading}}{\text{µL injected}} \times \frac{5}{\text{dry wt. fraction}}$	mg/L	mg/Kg

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporting Units	
	Liquid	Solid
Gross Alpha, Gross Beta		
Gross α or β = $\frac{(\text{cpm sample} - \text{cpm background})\,(1000)}{(2.22)(E_1)(\text{sample volume in mL or sample mass in mg})}$	pCi/L	pCi/g
E_1 = counting efficiency		
Counting error = $\frac{(A + B)^{1/2}\,(1.96)\,(1000)}{(2.22)(E_1)(\text{sample volume in mL or sample mass in mg})}$		
A = gross counts/(count time in min)^2 B = background counts/(count time in min)^2		
Radium 226	Liquid	
$Ra\text{-}226 = \frac{C \times zt_3}{(2.22)(E)(V)(1 - e^{-zt_1})(e^{-zt_2})(1 - e^{-zt_3})}$	pCi/L	
C = net count rate E = calibration constant for system and scintiliation cell V = sample volume in liters t_1 = elapsed time (days) between first and second de-emanations, and z = 0.181 days^{-1} t_2 = elapsed time (h) between second de-emanation and counting, and z = 0.00755 h^{-1} t_3 = counting time (min), and z = 1.26x10^{-4} h^{-1} z = decay constant for Radon 222		

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporting Units
Radium 228	Liquid
$$Ra\text{-}228 = \frac{C \times zt_3}{(2.22)(E)(V)(R)(1 - e^{-zt_1})(e^{-zt_2})(1 - e^{-zt_3})}$$	pCi/L
C = net count rate E = counter efficiency for Actinium 228 V = sample volume in liters R = (fractional chemical yield of yttrium carrier (fractional chemical yield of barium carrier) z = decay constant for Actinium 228 (0.001884 min^{-1}) t_1 = ingrowth time (min) t_2 = time interval between first yttrium hydroxide precipitation and start of counting time t_3 = counting time interval (min)	
Tritium	pCi/L
$$\text{Tritium} = \frac{C}{2.22} \times \frac{1000}{\text{sample size (mls)}}$$	
C = Net count rate in disintegrations/minute	
Data for solid or semisolid samples are reported on a dry weight basis.	

12.5.2 Data Review

Laboratory analytical results are reviewed by a second analysts or a section supervisor. Prior to entering the reportable data into the LIMS, laboratory raw data have been reviewed, stamped, and signed to ensure that all of the method specifications have been met. This includes checking the extraction, digestion, distillation, and other preparation logs, as well as ensuring that all precision and accuracy requirements are addressed, and all steps of the analyses have been completed. If any problems arise during the analysis of the sample batch, it is the responsibility of the analyst and the section supervisor to bring this to the attention of the project manager, section manager, and QA manager through a written corrective action report.

The field/sampling manager is responsible for data review of all field-generated data. This includes verifying that all field descriptive data are recorded as per Section 6, that all field calibration requirements have been met as per Section 9, that all field QC data have met criteria given in Section 5, and that field data are entered accurately on worksheets.

Data flags are used on reports as needed to inform the project manager and the client of any additional information that might aid in the interpretation of the data. The data flagging system incorporates data qualifiers which are similar to flags specified in the Contract Laboratory Program protocols, as well as additional flags used to help explain batch specific events.

When data acquisition and reporting have been completed, the project manager reviews and prepares the final report. Because the project managers have extensive experience in evaluating analytical data, they have developed both objective and subjective techniques for data review. Each value reported is reviewed in the context of the respective environmental matrix and all available QC/QA data. Outliers or other abnormal values are carefully scrutinized, and samples are reanalyzed if the abnormalities cannot be explained. Where there are cases in which the results from spiked samples suggest interferences, attempts are made to remove the interferences, or alternate analytical procedures are used. If the interference problem cannot be resolved, the data are flagged and/or a narrative is included with the report.

12.5.3 Special Project or Data Package Review

If special handling and/or data packages are requested by the client, QA personnel also review the project report and the raw data. This includes checking that holding time requirements are met, checking calibrations, reviewing all quality control data and/or control charts, and initiating any corrective action or reanalyses that might be appropriate.

12.6 Reporting

The final report is printed and signed by the project manager after all review has been completed. The data flags that may appear in a project report are defined on the signature page, and any additional comments are also footnoted on this page.

If requested by the client or a project specific QA Plan, custom reports or CLP data packages with diskette deliverables can be provided. If data packaging is requested, a paginated data package is provided in addition to the project report. The format of the project report and/or data package can be adjusted to meet the needs of the client. All LIMS reports can be downloaded onto diskettes or to most clients' computers.

12.7 Data Storage

The procedures and policies for raw data retention are described in SL SOP QC16: *Analytical Records Maintained by SL* and summarized briefly as follows. After the projects are completed, the data are transferred to a secured area and filed chronologically by laboratory section in boxes and maintained for five years or the term specified in a client contract. In cases where data are reviewed on a computer screen, and a tape back-up system is available, electronic files of data are stored on tape in lieu of paper data for a period of ten years. If the data are to be purged to the client or need to be separated from the general raw data files, the data can be boxed, labeled and stored in a separate secured area. Keys to the data storage areas are retained by the QA staff and the section/department managers.

All in-lab data generated by computer systems are stored to tape or on hard disk, when the capability exists. The tapes are labeled and stored at the individual work stations or maintained by a data systems manager and serve as the lab's raw data files.

Hard copies of all LIMS reports are maintained for five years in client files. All LIMS reports and associated QC data are kept for a minimum of three years on the LIMS hard diskettes and/or magnetic tape. All data on the LIMS are backed up daily on magnetic tape.

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13.0 NONCONFORMANCE AND CORRECTIVE ACTION PROCEDURES

A nonconformance is defined as any occurrence that prevents the lab from delivering data that are compliant with the control criteria published or incorporated by reference in an applicable quality assurance plan. The non-conformance report (NCR) form (Figure 13.1) is used to document nonconformance conditions and to specify the necessary action(s) taken to correct the specific problem. The corrective action report (CAR) form (Figure 13.2) is used in situations where a recurring problem or breakdown in systems is observed and warrants a more thorough investigation than a single event NCR. CARs may be initiated from:

- a specified NCR
- an observed trend or frequency of events that warrant corrective action
- an audit finding

Savannah Laboratories will abide by all reasonable corrective actions generated from documented findings by agency audits

Some situations that develop and require formal documentation may not be appropriate for an NCR and CAR. Some of these anomalous situations are detailed on the Anomaly Report (Figure 13.3).

The status of all NCRs and CARs are tracked in registries located in various departments or centrally located in the lab. Summaries of the NCRs and CARs are provided to the Lab Director and management periodically so that overall trends in nonconformances and corrective actions can be evaluated. The procedures for preparation, tracking and disposition of NCRs, CARs, and Anomaly Reports are given in SL SOP CA85: *Nonconformance and Corrective Action Procedures.*

Table 13.1 summarizes the checks, the acceptance criteria, and the recommended corrective action for various QC activities. This table and SL SOP AN02: *Analytical Batching* are used to evaluate sample and batch QC.

FIGURE 13.1

Nonconformance Report (NCR)

Initiated by:_____ Date initiated:_____ Client:_____ NCR #:_____

Issuing department/(division): _____ Project manager/(division) :_____ Method:_____

Project/SDG#:_____ Sample(s) affected:_____ Batch ID:_____

1	Nonconformance condition: Indicate and describe details.(if necessary) Describe reason problem occurred (root cause):	☐ Holding time ☐ Catastrophic Failure ☐ LCS/MS ☐ Method blank ☐ Calibration ☐ Internal standards ☐ Surrogates ☐ Other
2	Action taken (add details if necessary): PM Initials/date: TM/DM/PS Initials/date:	☐ Proceed with analysis ☐ Reprep/reanalyze ☐ Do not analyze ☐ Case narrative (discuss in Sect. 5) ☐ No action taken (justify)
3	Result of action taken in 2, above (add details if necessary):	☐ Reprep/reanalysis acceptable ☐ Reprep/reanalysis agrees w/ original ☐ Other
4	Case Narrative Comments (if necessary) Initials/date:	
5	Close-out: Additional corrective action required to prevent recurrence? ☐ YES ☐ NO If YES, initiate corrective action report (CAR): CAR Initiated #: _____ Closed by: Date:	
6	Original to TM/DM; Copy to PM; Copy to RF (QC level III/IV only)	

FANC34:12.03.96:0

FIGURE 13.2

Corrective Action Report (CAR)

CAR #:_____ Initiated by/date:_____

Lab Director attn. requested? ☐ Yes ☐ No (*if yes, copy LD after completing section 1, below*)

1	Responsible TM/DM - summarize non-conformance incident and comments:
2	State root cause Initials: Date:
3	CORRECTIVE ACTION Assigned To:_____Target completion date:_____ Corrective action has been completed on (date):_____ By (Initials):
4	QA Department Comments By (Initials): Date:
5	Follow-up dates / comments (if no comments, indicate as "none") Initial 2 week 2 month ☐ Add to internal systems checklist?

FIGURE 13.3

ANOMALY REPORT

Date: _____	Log #: _____	Sample ID: _____	Client: _____

Dept: ___EX ___GE ___LC ___ME ___RA ___CU ___SG ___SM ___VG ___VM ___AI	Analysis: _____	Reported by: _____

Anomaly:

- Sample matrix is different than indicated by log-in.

Logged in as	*Best described as*
Water	*Water*
Soil	*Non-aqueous liquid*
Oil	*Soil*
	Sludge
	Oil
	Product
	Other _____

- Sample was received with inadequate preservation, and was preserved upon receipt.
- Sample received in an incompatible sample container. ____ *glass* ____ *plastic* ____ *other* _____
- MS/MSD failed while the LCS/LCSD passed criteria, for a drinking water parameter. Method indicates data flagging.
- Target analyte(s) detected in drinking water sample. (Describe below)
- Sample exhibits gross non-homogeneity. (Describe below)
- Insufficient sample received for analysis.
- Data flag may be needed. Discuss with DM/LM before reporting.

Other _____

Custody: ***ALWAYS ATTACH A COPY OF COC WITH HIGHLIGHTED DEFICIENCY**

- Sample description discrepancy between COC & Container
- Sample container breakage
- Cooler temp >6EC or frozen
- Sample received not listed on COC
- Custody seals broken
- Incomplete COC
- Sample container partially filled
- Improperly preserved sample

Comments:	Client Notified: -Yes -No Contact:
	Date:
	Resolution:

Route to:
Division PM:

Other Div. PM: SL_____ ML_____ NL_____ FL_____ TL_____ BL_____

FAN038:04.09.97:3

TABLE 13.1CORRECTIVE ACTION

QC Activity	Acceptance Criteria	Recommended Corrective Action
GC/MS tuning	Section 9.0	Do not analyze samples unless criteria are met.
Initial calibration standards	Section 9.0	Reanalyze standards. If still unacceptable, remake standards or instrument corrections.
Continuing calibration standard	Section 9.0	Reanalyze standard. If still unacceptable, remake standards, or recalibrate.
Calibration blanks	< RL or QAPP/Method-defined criteria (for CLP procedures, use SOW guidelines)	Reanalyze calibration blank. If problem, determine source of contamination and reanalyze. Re-calibration may be required..
Method blank	< RL or QAPP/ Method-defined criteria (for CLP procedures, use SOW guidelines)	Reanalyze method blank. If problem, determine source of contamination. If necessary or possible, re-prep and re-analyze. Do not re-prep and re-analyze if no sample in batch or report contains the analyte(s) detected in the method blank. For SW-846 analyses, do not reanalyze if the method blank level is less than 5% of the regulatory limit or less than 5% of the lowest sample concentration .
Surrogate recovery (GC/MS semivolatiles)	Tables 5.1 - 5.8, or program/ project specific. One acid and one base may be out of criteria, provided all surrogates are in control in LCS	Follow method guidelines.
Surrogate recovery (GC/MS volatiles)	Tables 5.1 - 5.8, or program/project specific	Follow method guidelines.
Surrogate recovery GC or LC	Tables 5.1 - 5.8, or program/project specific	Check for possible matrix interferences or other causes and follow method guidelines.
Matrix spike recoveries	Tables 5.1 - 5.8, or program/project specific	Check for possible matrix interferences or other causes. If still out, evaluate LCS.(1)
Lab control standard (LCS) recoveries	Tables 5.1 - 5.8, or program/project specific	Check calculations, reanalyze standards, and if necessary or possible, redigest or extract batch and reanalyze.(1)
Precision of MS/MSD or sample duplicate	Tables 5.1 - 5.8, or QAPP Specific (used for evaluation but not control unless specified by the method)	Check calculation. Check for possible matrix interference or other causes.
Internal standards (organics)	Method or protocol-required limits	Follow method or protocol guidelines.
Trip blanks	≤ RL	Check related method blank for contamination.
Field Blanks	≤ RL	Check related method blank for contamination.
Equipment blanks	≤ RL	Check related method blank for contamination.
Field duplicates	Follow project/program requirements	Follow project/program requirements
Microbiology + and - controls for media	Should be + and -, respectively	Reject medium.
Microbiology duplicates	RPD with established limits	Follow agency requirements.

(1) See Section 11.2.1 for guidance on full list spikes and unstable compounds

TABLE 13.1CORRECTIVE ACTION

QC Activity	Acceptance Criteria	Recommended Corrective Action
Sample results	Calibration	If the calibration fails for a target and the corresponding target is not detected, the results may be reported as < RL if the RL standard is analyzed and detected.
	Spike criteria limits	If a limited list MS or LCS is high biased and no targets are detected above the RL, results are reported as < RL. When a full compound spike is utilized, and the MS or LCS result is high biased, and the corresponding target is not detected, the result for the corresponding target is reported as <RL, regardless of the other targets.
	Surrogate criteria limits	If surrogate recovery is high biased and no target is detected, the results are reported as <RL.
External Quality control check samples	Defined by the program or project.	Defined by the program or project.

14.0 PERFORMANCE SYSTEM AUDITS

14.1 Internal System Audits

14.1.1 Laboratory Audits

Annual laboratory audits are conducted by the division QA Manager or QA Officer. The scope and depth of the audit are determined according to the requirements of the division. The system audit includes, but is not limited to:

- evaluation of the procedures and items listed on the audit checklist
- review of compliance with the SL SOPs
- review of the compliance with this quality assurance plan
- review of the training records
- review of the nonconformance and anomaly reports and follow up on corrective actions from previous audits, external audits, or PE samples.

Some items may require more frequent auditing to determine if non-compliant procedures have been corrected. The internal audit may be performed quarterly for one or two sections with the goal of auditing of all systems once per year.

A report of the internal systems audit is prepared and submitted to the lab director and to the Corporate QA Manager by the end of January.

14.1.2 Corporate Systems Audit

A systems audit of each division is conducted annually by the Corporate QA Manager to determine if the procedures implemented by the SL divisions are in compliance with this quality assurance plan and the standard operating procedures (SOPs). This is primarily accomplished by the review of the following:

- the annual systems audit performed by the division QA Manager or QA Officer
- the findings and responses to external audits
- the results of PE samples
- summaries of nonconformance and corrective action taken by the lab.

The annual systems audit is performed by the Corporate QA Manager by the end of February. The Corporate QA Manager may request additional information or documentation of implementation. If necessary, the Corporate QA Manager will schedule an on-site evaluation of the division laboratories.

14.1.3 Field Audits

An audit of the field sampling procedures is performed annually. These systems audits are conducted by the QA Officer, an external auditor, or a regulatory agency. The audit includes all aspects of field sampling operations. Section 6.0 of this document defines the elements that serve as a basis for this audit.

14.2 External System Audits

Each laboratory may be certified by a number of state agencies, governmental agencies or private certification programs. Most of these programs require continuing on-site system audits of the laboratory. The laboratories submit to these on-sites as required by these certifying agencies and organizations and respond to any noted nonconformances with corrective actions.

Field system audits are performed periodically by various federal and state regulatory agencies. Field sampling and documentation procedures are examined to ensure sampling is performed according to the agency protocols.

14.3 Performance Audits

14.3.1 Internal Performance Audits

Internal performance audits or evaluations are routinely performed by Savannah Laboratories. Single blind performance audits are employed for several reasons. One purpose is to provide corrective action for parameters judged to be unacceptable on WP, WS or other major external performance audits. Periodic internal performance audits are also used to test parameters that are not routinely tested by external performance audits. Finally, single blind performance audits are employed to satisfy certain certification requirements, to satisfy auditors' specific requests for performance audit samples, or provide additional evidence of data quality to clients with specific questions regarding laboratory performance.

14.3.2 External Performance Audits

All facilities participate in each of the following performance evaluation audits semiannually:

1. U.S. EPA Water Supply Study (WS Series).
2. U.S. EPA Water Pollution Study (WP Series).

All facilities participate on an annual basis in a microbiology proficiency testing program.

Additionally, the laboratories participate in several regulatory agency, certifying group, or client requested performance audits. These performance audits include both single and double blind P.E. samples. Internal performance audits are logged into the LIMS system and analyzed and reported in the same manner as samples. Results from these performance audits are reported to management, agencies, and clients as required. Nonconformance Reports (NCR) and Corrective Action Reports (CAR) are issued when appropriate.

Results from agency performance audits are supplied to clients upon request.

15.0 QUALITY ASSURANCE REPORTS

15.1 Internal Reports

The QA Officer or QA Manager of each division is responsible for providing quality assurance reports to the division Lab Director and to the Corporate QA Manager on an annual basis. The report must include the following elements:

- complete internal audit checklist
- summaries of nonconformance/corrective actions from routine lab operations
- finding and responses to external audits and PE samples with any nonconformance/corrective action initiated
- results from all PE samples (internal and external)

These annual reports will be summarized by the Corporate QA Manager and provided to the President and Vice Presidents for review by the end of March.

15.2 External Reports

Quality assurance or program reports are made to all required agencies or offices. The scope, content, and frequency of these reports are generally defined by the agency or office.

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16.0 TRAINING AND QUALIFICATIONS

Savannah Laboratories' greatest asset is its well qualified and trained staff. The duties and responsibilities of management and staff positions are described in Section 4.0 of this manual. In addition, the qualifications for each of these positions can be obtained from the Corporate Human Resources Director at the Savannah Division.

SL SOP CA01 (*SL Training SOP)* describes the procedures and documentation required to adequately train the analytical staff. All new employees are required to undergo an introduction to Savannah Labs' policies described in SL SOP CA10: *Procedures for New Employee Orientation.*

17.0 DOCUMENTS AND RECORDS

All documentation and records are maintained in accordance with SL SOPs AN45:*Laboratory Notebooks,* CA02:*Divisional Document Control and Distribution,* and CA80:*Data Generation, Entry, Review, Approval, and Reporting..*

Section 12 (Data Reduction, Review, and Reporting) of this manual describes the flow of data through the laboratory and the SL policy for document and record retention (Section 12.7). SL SOP QC16: *Analytical records Maintained by SL* describes the policies and procedures for the retention of analytical records (raw data and associated QC data).

18.0 PROCUREMENT

The SL policies and procedures for procurement are described in SL SOP CA 45: *Procurement.*

19.0 QUALITY IMPROVEMENT/MANAGEMENT ASSESSMENT

Savannah Laboratories and Environmental Services, Inc., is committed to quality improvement and customer service. All aspects of the laboratories operations are monitored and input from clients is evaluated to determine if present policies and procedures are meeting the objectives defined in Section 3.0 of this manual. The following SOPs have been implemented to address and document quality improvement:

Procurement of Laboratory Materials-SL SOP CA45: *Procurement*
Analytical Training- SL SOP CA01: *SL Training SOP*
Client Satisfaction and Complaint Resolution - SL SOP CA95: *Complaint Resolution*
Non-Conformance and Corrective Action - SL SOP CA85:*Noncomformance and Corrective Action Procedures*
Auditing (Divisional and Corporate) - SL SOP CA05:*Technical and Systems Audits (Divisional and Corporate)*

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Semivolatiles - GC/MS	Target Compound List[1]	Priority Pollutant List[2]	Appendix IX List[3]
Acenaphthene	X	X	X
Acenaphthylene	X	X	X
Acetophenone			X
2-Acetylaminofluorene			X
4-Aminobiphenyl			X
Aniline			X
Anthracene	X	X	X
Aramite			X
Benzidine		X	
Benz(a)anthracene	X	X	X
Benzo(b)fluoranthene	X	X	X
Benzo(k)fluoranthene	X	X	X
Benzo(g,h,i)perylene	X	X	X
Benzo(a)pyrene	X	X	X
Benzyl alcohol			X
Bis(2-chloroethoxy)methane	X	X	X
Bis(2-chloroethyl)ether	X	X	X
Bis(2-chloroisopropyl) ether/2,2'-Oxybis(1-Chloropropane)	X	X	X
Bis(2-ethylhexyl)phthalate	X	X	X
4-Bromophenyl phenyl ether	X	X	X
Butyl benzyl phthalate	X	X	X
Carbazole	X		
4-Chloroaniline	X		X
4-Chloro-3-methylphenol	X	X	X
2-Chloronaphthalene	X	X	X
2-Chlorophenol	X	X	X
4-Chlorophenyl phenyl ether	X	X	X
Chrysene	X	X	X
Diallate			X
Dibenz(a,h)anthracene	X	X	X
Dibenzofuran	X		X
Di-n-butylphthalate	X	X	X
1,2-Dichlorobenzene	X	X	X
1,3-Dichlorobenzene	X	X	X
1,4-Dichlorobenzene	X	X	X
3,3-Dichlorobenzidine	X	X	X
2,4-Dichlorophenol	X	X	X
2,6-Dichlorophenol			X
Diethylphthalate	X	X	X

Volatile-GC Method 8021	Target Compound List[1]	Priority Pollutant List[2]	Appendix IX List[3]
Benzene	NA	X	NA
Bromodichloromethane	NA	X	NA
Bromoform	NA	X	NA
Bromomethane	NA	X	NA
Carbon Tetrachloride	NA	X	NA
Chlorobenzene	NA	X	NA
Chlorodibromomethane	NA	X	NA
Chloroethane	NA	X	NA
2-Chloroethylvinyl ether	NA	X	NA
Chloroform	NA	X	NA
Chloromethane	NA	X	NA
1,2-Dichlorobenzene	NA	X	NA
1,3-Dichlorobenzene	NA	X	NA
1,4-Dichlorobenzene	NA	X	NA
1,1-Dichloroethane	NA	X	NA
1,2-Dichloroethane	NA	X	NA
1,1-Dichloroethene	NA	X	NA
cis-1,2-Dichloroethene	NA	(*)	NA
trans-1,2-Dichloroethene	NA	X	NA
1,2-Dichloropropane	NA	X	NA
cis-1,3-Dichloropropene	NA	X	NA
trans-1,3-Dichloropropene	NA	X	NA
Ethylbenzene	NA	X	NA
Methylene Chloride (Dichloromethane)	NA	X	NA
Methyl tert-butyl ether (MTBE)	NA	(*)	NA
1,1,2,2-Tetrachloroethane	NA	X	NA
Tetrachloroethene	NA	X	NA
Toluene	NA	X	NA
1,1,1-Trichloroethane	NA	X	NA
1,1,2-Trichloroethane	NA	X	NA
Trichloroethene	NA	X	NA
Trichlorofluoromethane	NA	X	NA
Vinyl Chloride	NA	X	NA
Xylenes, total	NA	(*)	NA

NA-The volatiles for the TCL and Appendix IX Lists are routinely analyzed by GC/MS method 8260.

(*) These compounds are not on the Priority Pollutant List, but are routinely reported when method 8021 is used.

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 02/07/97 Zeeman Cu SW846/200 series
BATCH CU01 MDL'S

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	15.0

PROGRAM 17 Cu NO MODIFIER

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	1
LAMP CURRENT (mA)	4
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	327.4
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.50

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	135	15.0	3.0	NORMAL	NO
2	200	10.0	3.0	NORMAL	NO
3	300	5.0	3.0	NORMAL	NO
4	500	5.0	3.0	NORMAL	NO
5	750	15.0	3.0	NORMAL	NO
6	750	3.0	1.0	NORMAL	NO
7	2100	1.0	0.0	NORMAL	YES
8	2100	2.0	0.0	NORMAL	YES
9	2600	1.0	3.0	NORMAL	NO
10	45	13.3	3.0	NORMAL	NO

02.07.97

SAMPLER PARAMETERS

VOLUMES (µL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	20	
STANDARD 1	20	0	
STANDARD 2	20	0	
STANDARD 3	20	0	
STANDARD 4	20	0	
SAMPLE	20	0	

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 75
INJECT RATE 7

QC PROTOCOL PARAMETERS

Working Standard Lot # XS-Z

Date Prepared 02.07.97

Analyst LR

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (µL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (µL)	8
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	0.3 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS
BLANK QC	0.0		0.054	0.092 0.016 07Feb97 07:37 CONCENTRATION LESS THAN IDL
BLANK QC	0.0		0.003	0.011 -0.004 07Feb97 07:43 CONCENTRATION LESS THAN IDL
BLANK QC	0.0		-0.000	0.005 -0.005 07Feb97 07:47 CONCENTRATION LESS THAN IDL
STANDARD 1 QC	5.0	0.0	0.026	0.026 0.026 07Feb97 07:51
STANDARD 2 QC	10.0	2.3	0.058	0.057 0.059 07Feb97 07:55
STANDARD 3 QC	50.0	0.8	0.303	0.301 0.305 07Feb97 07:59

TJA Video 22 Ni SW846/200 series

Atomizer: Furnace
M- -ix: ULTRAPURE WATER

METHOD INFORMATION **

Default Setup:

Number of Repeats : 2
Flush Time (sec) : 0.0
Auto-Increment Sample Names? No

Auto-print Calibration Curves? : No
Analysis Graphics Display : Aborbances & Temp Profile
Auto-print Analysis Graphics? : No
Auto-save Analysis Graphics? : No

Default File Names:

Analysis Data File	: NI	Sample Limits Table	: LCTAB
Autosampler Table	: AUTOTAB	Blank (AZ) Limits Table	: BLANKLCT
		QC Check Table	: QCTAB
		Recovery Check Table	: RQCTAB

)' TT INFORMATION **

) ut Mode: Concentration
)verride Print Limits? Yes
)verride Significant Figures? No
.imits Table: LCTAB Check? No
:orrection Factor: 1

.uto-print data? Yes
:ondensed report format? Yes
.uto-store data? Yes
.tore individual repeats? Yes

:eport to:
Screen Avgs, Stats, Reps, Checks, Units
Printer Avgs, Stats, Reps, Checks, Units

mm/LL

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR/MM
DATE 02/12/97
BATCH CD03 0211J,MDL'S

Zeeman Cd SW846/200 series

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 29 Cd AMM.PHOS.

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	3
LAMP CURRENT (mA)	4
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	228.8
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.00

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	180	15.0	3.0	NORMAL	NO
2	250	10.0	3.0	NORMAL	NO
3	500	20.0	3.0	NORMAL	NO
4	500	10.0	3.0	NORMAL	NO
5	500	2.0	0.1	NORMAL	NO
6	2000	1.0	0.1	NORMAL	YES
7	2000	3.0	0.1	NORMAL	YES
8	2500	1.0	3.0	NORMAL	NO
9	45	12.3	3.0	NORMAL	NO

02.12.
mm/L

SAMPLER PARAMETERS
VOLUMES (ΛL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	15	4
STANDARD 1	15	0	4
STANDARD 2	15	0	4
STANDARD 3	15	0	4
STANDARD 4	15	0	4
SAMPLE	15	0	4

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 110
INJECT RATE 7

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (ΛL)	15
QC STANDARD CONCENTRATION	5.00 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (ΛL)	6
QC SPIKE CONCENTRATION	5.00 ug/L
MATRIX SPIKE CONCENTRATION	5.00 ug/L
INSTRUMENT DETECTION LIMIT	0.06 ug/L
REQUIRED DETECTION LIMIT	1.00 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.00		0.333	0.665	0.001
QC				12Feb97	15:07
				CONCENTRATION LESS THAN IDL	
BLANK	0.00		0.001	0.001	0.001
QC				12Feb97	15:13
				CONCENTRATION LESS THAN IDL	
STANDARD 1	1.00	4.8	0.031	0.032	0.030
QC				12Feb97	15:17
STANDARD 2	2.00	0.5	0.061	0.061	0.060
QC				12Feb97	15:22
STANDARD 3	5.00	2.0	0.147	0.145	0.149
				12Feb97	15:26
STANDARD 4	10.00	0.3	0.276	0.276	0.277
QC				12Feb97	15:31

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR Zeeman Ag SW846/200 series
DATE 02/24/97
BATCH AG03 MDL'S

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 27 Ag AMM.PHOS.

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	2
LAMP CURRENT (mA)	4
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	328.1
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.30

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	120	20.0	3.0	NORMAL	NO
2	200	10.0	3.0	NORMAL	NO
3	800	15.0	3.0	NORMAL	NO
4	800	5.0	3.0	NORMAL	NO
5	800	2.0	0.1	NORMAL	NO
6	2400	0.9	0.0	NORMAL	YES
7	2400	2.0	0.0	NORMAL	YES
8	2600	3.0	3.0	NORMAL	NO
9	45	13.3	3.0	NORMAL	NO

02-24-97
CL

SAMPLER PARAMETERS
VOLUMES (fL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	20	2
STANDARD 1	20	0	2
STANDARD 2	20	0	2
STANDARD 3	20	0	2
STANDARD 4	20	0	2
SAMPLE	20	0	2

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 100
INJECT RATE 9

QC PROTOCOL PARAMETERS

Working Standard Lot # IT-2
Date Prepared 02-24-97
Analyst CL

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (fL)	20
QC STANDARD CONCENTRATION	5.00 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	8
QC SPIKE CONCENTRATION	5.00 ug/L
MATRIX SPIKE CONCENTRATION	5.00 ug/L
INSTRUMENT DETECTION LIMIT	0.03 ug/L
REQUIRED DETECTION LIMIT	1.00 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.00		0.001	0.001	0.000
QC				24Feb97	12:56
				CONCENTRATION LESS THAN IDL	
STANDARD 1	1.00	3.0	0.040	0.039	0.041
QC				24Feb97	13:01
STANDARD 2	2.00	0.0	0.082	0.082	0.082
QC				24Feb97	13:05
STANDARD 3	5.00	0.4	0.204	0.204	0.205
QC				24Feb97	13:10
STANDARD 4	10.00	0.7	0.389	0.387	0.391
QC				24Feb97	13:15

Atomizer: Furnace
Matrix: SOIL AND WASTE WATER TJA 4000 As/Se SW846/200 series

THOD INFORMATION **

Default Setup:

Number of Repeats : 2
Flush Time (sec) : 5.0
Auto-Increment Sample Names? No

Auto-print Calibration Curves? : Yes
Analysis Graphics Display : Aborbances & Temp Profile
Auto-print Analysis Graphics? : No
Auto-save Analysis Graphics? : No

Default File Names:

Analysis Data File : AFEB971
Autosampler Table : AUTOTAB
Sample Limits Table : LCTAB
Blank (AZ) Limits Table : BLANKLCT
QC Check Table : QCTAB
Recovery Check Table : RQCTAB

OUTPUT INFORMATION **

put Mode: Concentration
rride Print Limits? Yes
Override Significant Figures? No
Limits Table: LCTAB Check? No
Correction Factor: 1

Auto-print data? Yes
Condensed report format? Yes
Auto-store data? Yes
Store individual repeats? Yes

Report to:
Screen Avgs, Stats, Reps, Checks, Units
Printer Avgs, Stats, Reps, Checks, Units

Elements : As Se

Mode : Single Beam Absorption

Integ Type : Peak Area Integ Time : 1.5 Sec. Delay Time : 1.2 Sec.
Graph Scale: Automatic
Comment : 0.1 mL 1.0% Ni to 5mL Sample - PLATFORM TUBE

Furnace Information:

	Dry	Pyro1	Pyro2	Atom	Clean
Temp	200	900	900	2600	2600
Ramp	3	15	0	0	-
Hold	0	10	2	4	2
Purge	Low	Medium	Low	Off	High

Integ Stage : Atom Air Ash : Off Auto-Baseline Corr.? Yes Economizer? No
Deposit Time: 12.0 Delay Time: 15 Auto-Deposit Adjust? No

Element : As
Element Name : As
Wavelength : 197.20
Bandpass : 1.0
Lamp Pos. : 1
High Voltage : Manual Value: 600
BKG Method : SH
Lamp Current : norm: 3.0 bkg: 2.5
Signif Figs : 5
Print Units : ppb
Print Limits Low : 0
Print Limits High: 0

Stdzn Method: Multipoint Stdn.

	Std Names	Std Conc	Abs.
A/Z#1:	BlankSTD	= 0.00	= 0.0000
#2:	5.00STD	= 5.00	= 0.0110
#3:	10.00STD	= 10.00	= 0.0250
A/C#4:	50.00STD	= 50.00	= 0.1193
#5:	100.00STD	= 100.00	= 0.2243

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

Element : Se
Element Name : Se
Wavelength : 196.03
Bandpass : 1.0
Lamp Pos. : 2
High Voltage : Manual Value: 642
BKG Method : SH
Lamp Current : norm: 3.4 bkg: 2.0
Signif Figs : 5
Print Units : ppb
Print Limits Low : 0
Print Limits High: 0

Stdzn Method: Multipoint Stdn.

	Std Names	Std Conc	Abs.
A/Z#1:	BlankSTD	= 0.00	= 0.0000
#2:	5.00STD	= 5.00	= 0.0102
#3:	10.00STD	= 10.00	= 0.0183
A/C#4:	50.00STD	= 50.00	= 0.0918

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

'Atomizer: Furnace
Matrix: ULTRAPURE WATER TJA 4000 As/Se SW 846/200 series

.THOD INFORMATION **

Default Setup:

Number of Repeats : 2
Flush Time (sec) : 8.0
Auto-Increment Sample Names? No

Auto-print Calibration Curves? : Yes
Analysis Graphics Display : Aborbances & Temp Profile
Auto-print Analysis Graphics? : No
Auto-save Analysis Graphics? : No

Default File Names:

Analysis Data File : AJAN972
Autosampler Table : AUTOTAB
Sample Limits Table : LCTAB
Blank (AZ) Limits Table : BLANKLCT
QC Check Table : QCTAB
Recovery Check Table : RQCTAB

OUTPUT INFORMATION **

tput Mode: Concentration
erride Print Limits? Yes
override Significant Figures? No
Limits Table: LCTAB Check? No
Correction Factor: 1

Auto-print data? Yes
Condensed report format? Yes
Auto-store data? Yes
Store individual repeats? Yes

Report to:
Screen Avgs, Stats, Reps, Checks, Units
Printer Avgs, Stats, Reps, Checks, Units

Elements : As Se

Mode : Single Beam Absorption

Integ Type : Peak Area Integ Time : 2.2 Sec. Delay Time : 0.6 Sec.
Graph Scale: Automatic
Comment : 1%PD/2%MG NONPLATFORM TUBE

Furnace Information:

	Dry	Pyro1	Pyro2	Atom	Clean
Temp	150	900	890	2200	2600
Ramp	2	15	1	0	-
Hold	0	10	1	4	3
Purge	Medium	Medium	Low	Off	High

Integ Stage : Atom Air Ash : Off Auto-Baseline Corr.? Yes Economizer? No
Deposit Time: 10.0 Delay Time: 12 Auto-Deposit Adjust? No

Element : As
Element Name : As
Wavelength : 197.20
Bandpass : 1.0
Lamp Pos. : 1
High Voltage : Manual Value: 621
BKG Method : SH
Lamp Current : norm: 3.4 bkg: 2.5
Signif Figs : 5
Print Units : ppb
Print Limits Low : 0
Print Limits High: 0

Stdzn Method: Multipoint Stdn.

	Std Names	Std Conc	Abs.
A/Z#1:	BlankSTD	= 0.00	= 0.0000
#2:	5.00	= 5.00	= 0.0092
#3:	10.00	= 10.00	= 0.0183
A/C#4:	50.00	= 50.00	= 0.0941
#5:	100.00	= 100.00	= 0.1834

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

Element : Se
Element Name : Se
Wavelength : 196.00
Bandpass : 1.0
Lamp Pos. : 2
High Voltage : Manual Value: 645
BKG Method : SH
Lamp Current : norm: 3.4 bkg: 2.2
Signif Figs : 5
Print Units : ppb
Print Limits Low : 0
Print Limits High: 0

Stdzn Method: Multipoint Stdn.

	Std Names	Std Conc	Abs.
A/Z#1:	BlankSTD	= 0.00	= 0.0000
#2:	5.00	= 5.00	= 0.0079
#3:	10.00	= 10.00	= 0.0152
A/C#4:	50.00	= 50.00	= 0.0770

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

mizer: Furnace
rix: 1 % HNO3 IN DI WATER

TJA Video 22 Pb/Tl SW846, 200 series

METHOD INFORMATION **

Default Setup:

Number of Repeats : 2
Flush Time (sec) : 7.0
Auto-Increment Sample Names? No

Auto-print Calibration Curves? : Yes
Analysis Graphics Display : Aborbances & Temp Profile
Auto-print Analysis Graphics? : No
Auto-save Analysis Graphics? : No

Default File Names:

Analysis Data File : BFEB971
Autosampler Table : AUTOTAB
Sample Limits Table : LCTAB
Blank (AZ) Limits Table : BLANKLCT
QC Check Table : QCTAB
Recovery Check Table : RQCTAB

UT INFORMATION **

Output Mode: Concentration
Override Print Limits? Yes
Override Significant Figures? No
Limits Table: LCTAB Check? No
Correction Factor: 1

Auto-print data? Yes
Condensed report format? Yes
Auto-store data? Yes
Store individual repeats? Yes

Report to:
Screen Avgs, Stats, Reps, Checks, Units
Printer Avgs, Stats, Reps, Checks, Units

Elements : Pb Tl

Mode : Single Beam Absorption

Integ Type : Peak Area Integ Time : 3.0 Sec. Delay Time : 0.9 Sec.
Graph Scale: Automatic
Comment : MODIFIER 10 % NH4(PO4)/MATRIX 1 % HNO3

Furnace Information:

	Dry	Pyro1	Pyro2	Atom	Clean
Temp	150	350	650	2100	2600
Ramp	10	10	15	0	-
Hold	0	0	5	3	3
Purge	Low	Medium	Medium	Off	High

Integ Stage : Atom Air Ash : Off Auto-Baseline Corr.? Yes Economizer? No
Deposit Time: 8.0 Delay Time: 10 Auto-Deposit Adjust? No

Element : Pb
Element Name : Pb
Wavelength : 283.30
Bandpass : 0.50
High Voltage : 700
BKG Method : SH
Lamp Current : norm: 2.0 bkg: 1.5
Signif Figs : 5
Print Units : ppb
Print Limits Low : 0
Print Limits High: 0

Stdzn Method: Multipoint Stdn.

Std Names	Std Conc	Abs.
A/Z#1: BlankSTD	= 0.00	= 0.0000
#2: 3.00	= 3.00	= 0.0169
#3: 10.00	= 10.00	= 0.0515
A/C#4: 50.00	= 50.00	= 0.2496
#5: 100.00	= 100.00	= 0.4614

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

Element : Tl
Element Name : Tl
Wavelength : 276.80
Bandpass : 0.50
High Voltage : 690
BKG Method : SH
Lamp Current : norm: 2.5 bkg: 2.0
Signif Figs : 5
Print Units : ppb
Print Limits Low : 0
Print Limits High: 0

Stdzn Method: Multipoint Stdn.

Std Names	Std Conc	Abs.
A/Z#1: BlankSTD	= 0.00	= 0.0000
#2: 3.00	= 3.00	= 0.0106
#3: 10.00	= 10.00	= 0.0354
A/C#4: 50.00	= 50.00	= 0.1674
#5: 100.00	= 100.00	= 0.3048

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

CHANGE -IN-PROGRESS ATTACHMENT

SOP Document No: SM05:12.19.97:5

SOP Description: Semivolatile Compounds by GC/MS (8270C)

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager Date: 4/13/98

The following revisions or additions have been made to the referenced SOP.

Changes or updates to all sections are in **bold type**.

8.2.3 The following tables are examples of the preparation of the calibration standards. If the preparation steps in the following tables are not used, the lab must document the "recipe" used to prepare the standards in the traceability log. These recipes may change as different vendors are contracted and as more suitable solutions become available.

BNA Intermediate Standard (BNA-200)

STOCK STANDARD	STOCK CONC (ug/mL)	uL Stock	FVOL	Conc (ug/mL)
SV MIX 1	2000	0.50	5.0mL	200
SV MIX 2	2000	0.50		200
SV MIX 3	2000	0.50		200
SV MIX 4	2000	0.50		200
SV MIX 5	2000	0.50		200
SV MIX 7	2000	0.50		200
SV CUSTOM	2000	0.50		200
Pyridine	5000	0.20		200
605 BENZIDINE	2000	0.50		200
ACID SURROGATE	**10000**	**0.10**		**200**
B/N SURROGATE	**5000**	**0.20**		**200**

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

Restek RTX-5MS 30m x 0.25mm ID, 0.5um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45 C for 3 minutes
Column temperature program : 10C per minute
Final column temperature: 300C (until at least one minute past the elution time of Benzo(g,h,i) perylene).

GC injector parameters

Injector temperature : **250-280C**
Injector : splitless
Inlet purge time : 0.8 minutes
Injector liner : 4mm ID quartz or 4mm glass, deactivated
Sample injection volume : 1-2uL

Mass Spectrometer and interface parameters

Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: 35-500amu, **with a minimum scan time of 1.0 scans per second**

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. SL SOP AN02 also describes the procedure for evaluating batch-specific QC. The attached SOP summary and Table 13.1 in the SL Corporate QA Plan provide guidance for evaluating sample data.

Appendix-Example Quantitation Report

Quantification of Di-N-octyl phthalate

Di-N-octyl phthalate will be associated with the Chrysene-d12 internal standard. The quantitation report attached to the SOP lists Perylene-d12 as di-N-octyl phthalate's internal standard.

Approval Signature: R Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager Date: 12/19/97

SEMI-VOLATILE COMPOUNDS BY GC/MS (8270C)

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of various semi-volatile organic compounds (SVOC) in groundwater, TCLP leachates, soils, sediments, waste, and solid sample extracts. The attached quantitation report lists the target compounds, the retention times of the target compounds, the characteristic ions of the target compounds, and the internal standard association of each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for each target compound is given in Section 5 of the current revisions of the Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the mass spectra determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

This procedure is based on the guidance provided in SW-846 Method 8270C.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.2 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products such as diesel or waste oil.

4.3 Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ storage	Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C +/- 2C	1-L amber	7 days	40 days
Soil/ sediment	none; 4C +/- 2C	500-mL	14 days	40 days
Waste	none; 4C +/- 2C	Glass	14 days	40 days
TCLP	none; 4C +/- 2C	1-L amber	7 days from TCLP leaching procedure	40 days

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph- Hewlett-Packard (HP) 5890 or equivalent with compatible autosampler, splitless injector, and direct capillary interface

6.2 Mass spectrometer- HP 5970, HP5971, HP5972, or equivalent

6.3 Recommended Capillary column-Restek RTX-5MS, 30m x 0.25mm ID, 0.5um film thickness. Equivalent columns can be used.

6.4 Data system- compatible with GC/MS system

6.5 Microsyringes-

6.6 Volumetric flasks, Class A-appropriate volumes

6.7 Analytical balance

6.8 Autosampler vials and crimper, compatible with autosampler

7.0 REAGENTS

7.1 Methylene chloride-pesticide residue grade, for preparation of standards

7.2 Acetone-pesticide residue grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in SL SOP AN43:*Standard Preparation.*

The lab should purchase certified solutions from SL-approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SL SOP AN43 for guidance for standard preparation from neat materials.

8.1 Preparation of the Stocks from Neat Standards

The steps for the preparation of primary stock standards from neat materials are given in SL SOP *AN40: Standard Preparation and Traceability.* The standards should be prepared in methylene chloride but may require other solvents to dissolve the material.

8.2 Preparation the calibration standards from the stock standards

8.2.1 A minimum of five calibration standards are prepared. The concentrations of the stock standards are in the 1000-10000ug/mL range. The recommended standards are 10ug/mL, 40ug/mL, 60ug/mL, 80ug/mL, 100ug/mL, and 125ug/mL. The lowest level standard should be at the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve and seven calibration points are required for a polynomial regression curve (see SW-846 Method 8000B).

8.2.2 The following equation is used to determine the volume of a stock standard needed to prepare a given volume of a calibration standard:

$$Vs = \frac{Ccal \otimes Vcal}{Cs}$$

where

Vs = volume of stock standard required to prepare the calibration standard(mL)
Cs = concentration of the stock standard (ug/mL)
Ccal = concentration of the calibration standard to prepare (ug/mL)
Vcal = volume of calibration standard to prepare(mL)

EXAMPLE
Prepare 2mL of an 80ug/mL standard from a 2000ug/mL stock standard.

$$Vs = \frac{80ug/mL \otimes 2mL}{2000ug/mL} = 0.080mL = 80uL$$

NOTE: A primary concern when preparing calibration standards for BNA analysis is to make sure that all of the standards will "fit" in the volume of standard that will be prepared. As Table 2 demonstrates, the volumes of the various standards mixed is very near the capacity of the volumetric flask. Standards can be prepared using syringes to deliver the required volumes of standard and solvent directly to the storage vial.

8.2.3 The following tables are examples of the preparation of the calibration standards. If the preparation steps in the following tables are not used, the lab must document the "recipe" used to prepare the standards in the traceability log. These recipes may change as different vendors are contracted and as more suitable solutions become available.

BNA Intermediate Standard (BNA-200)

STOCK STANDARD	STOCK CONC (ug/mL)	uL Stock	FVOL	Conc (ug/mL)
SV MIX 1	2000	0.50	5.0mL	200
SV MIX 2	2000	0.50		200
SV MIX 3	2000	0.50		200
SV MIX 4	2000	0.50		200
SV MIX 5	2000	0.50		200
SV MIX 7	2000	0.50		200
SV CUSTOM	2000	0.50		200
Pyridine	5000	0.20		200
605 BENZIDINE	2000	0.50		200
ACID SURROGATE	2000	0.50		200
B/N SURROGATE	2000	0.50		200

BNA Calibration Standards

FINAL STANDARD	STOCK	CONC (ug/mL)	uL Stock	FVOL	Conc*
BNA-1	BNA-200	200	50	1.0	10
BNA-2	BNA-200	200	200	1.0	40
BNA-3	BNA-200	200	350	1.0	60
BNA-4	BNA-200	200	400	1.0	80
BNA-5	BNA-200	200	500	1.0	100
BNA-6	BNA-200	200	625	1.0	125

*After the standards are prepared, 20uL of the 2000ug/mL internal standard solution are added to each calibration standard.

APPENDIX IX (AP9) Intermediate Standard

STANDARD	CONC (ug/mL)	AP9-200
Custom Mix 1	2000	100
Custom Mix 2	2000	10
Custom Mix3	2000	100
Custom Mix 4	2000	100
Custom Mix 5	2000	100
Aramite	500	400

AP9 Calibration Standards

L STANDARD	Stock	CONC (ug/mL)	uL Stock	FVOL	Conc*
AP9-1	AP9-200	200	50	1.0	10
AP9-2	AP9-200	200	200	1.0	40
AP9-3	AP9-200	200	300	1.0	60
AP9-4	AP9-200	200	400	1.0	80
AP9-5	AP9-200	200	500	1.0	100
AP9-6	AP9-200	200	625	1.0	125

*After the standards are prepared, 20uL of the 2000ug/mL interanl standard solution are added to each calibration standard.

GC/MS Tuning and Column Evaluation Standard

STOCK STANDARD	CONC (ug/mL)	uL of stock to 1.0mL Methylene Chloride	ug/mL
SV Tuning Compound (DFTPP)	2500	20	50
Pentachlorophenol	2000	25	50*
p,p'-DDT	2000	25	50
605 Benzidines	2000	25	50**
SV ISTD(optional)	2000	20	40

*. standard level **below routine RL

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP Number	Extraction Technique
Aqueous, TCLP leachates	EX30	Continuous Liquid-liquid Extraction
Aqueous, TCLP leachates	EX35	Separatory Funnel
Soils/Sediments	EX40	Sonication
Wastes	EX42	Waste dilution

9.2 The sample concentration procedures are given in SL SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (GPC-SL SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products.

10.0 PROCEDURE

1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

Restek RTX-5MS 30m x 0.25mm ID, 0.5um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45 C for 3 minutes
Column temperature program : 10C per minute
Final column temperature: 300C (until at least one minute past the elution time of Benzo(g,h,i) perylene).

GC injector parameters

Injector temperature : 250-260°C
Injector : splitless
Inlet purge time : 0.8 minutes
Injector liner : 4mm ID quartz or 4mm glass, deactivated
Sample injection volume : 1-2uL

Mass Spectrometer and interface parameters

Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: 35-500amu, with a scan time of 1.0 scans per second or greater

Calibration

A minimum of five calibration standards are prepared The concentrations of the stock standards are in the 1000-10000ug/mL range. The recommended standards are 10ug/mL, 40ug/mL, 60ug/mL, 80ug/mL, 100ug/mL, and 125ug/mL. The lowest level standard should be at the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve and seven calibration points are required for a polynomial regression curve (see SW-846 Method 8000B).

The GC/MS system will be calibrated by the analysis of a minimum of five calibration standards with the following exception: target compounds that have reporting limits at 50ug/L (1700ug/kg) or above. These target compounds are generally nitro-substituted compounds with low sensitivities and erratic chromatographic behavior. The reasons for analyzing and evaluating fewer than five calibration standard for these compounds include:

1) the standard mixes contain all of the target compounds at the same concentrations
2) the sensitivities of these compounds are low
3) the sensitivities of the remaining target compounds are much higher. The difference between the sensitivities is such that a GC/MS system cannot easily or routinely be adjusted to allow for the detection of all of the target compounds in the BNA list at all five calibration levels
4) a standard at the RL is routinely analyzed and evaluated for these compounds and all positive hits for these compounds must fall within the limited range of detection.

Note: If the calbration points indicated above are used, it should be possible to analyze a minimum of 5 points for almost all compounds.

10.2.1 Fifty nanograms of DFTPP must be analyzed at the beginning of each 12 hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of DFTPP. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage.

10.2.1.1 Prepare a 50ng/uL solution of DFTPP column evaluation standard

?.1.2 Analyze a 1uL aliquot of the 50ng/uL DFTPP/column evaluation solution.

10.2.1.3 Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

-The following records must be kept for each DFTPP analysis that meets the criteria:
- the date, time, and data file of the analysis
- a spectrum of the scan or enhanced scans
- a tabulation of the ion abundances of the scan or enhanced scans

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

?.1.4 Benzidine and pentachlorophenol should be present at their normal responses with no peak tailing visible. This is a good check on the system: if pentachlorophenol (a CCC) does not respond well, the calibration standard should not be analyzed. Perform injector port and column maintenance and reanalyze the tune/column evaluation standard.

The p,p'- DDT breakdown is calculated using the following equation. The percent breakdown should not exceed 20%.

$$\%Breakdown = \frac{(areaDDE + areaDDD)}{(areaDDT + areaDDE + areaDDD)} x\ 100$$

Areas from the total ion chromatogram are evaluated to determine the DDT breakdown.

10.2.2 After the DFTPP criteria has been met, the initial calibration standards are analyzed

10.2.2.1 Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range. See section 8.2.1 for guidance regarding calibration levels.

10.2.2.2 Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts.

10.2.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.2.4 The relative response factor for each compound is calculated using the data system or using a PC spreadsheet as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See the attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

10.2.5 The average relative response factor (RRF_{avg}) is calculated for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3.... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

NOTE: As noted earlier, some target compounds may have fewer than five calibration standard levels. See section 8.2.1 and 10.2.

10.2.6 Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_l}}{} \frac{(RRF_i - RRF_{avg})^2}{n-1}}$$

10.2.7 Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\ 100$$

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in the SOP Summary. The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of sample extracts can begin.

If CCC and/or SPCC do not meet the stated criteria, all targets that are reported must meet the CCC criteria. For example, if naphthalene is the only target to be reported, it can be reported if 1) the CCC and SPCC pass; OR 2) naphthalene passes the CCC criteria (other targets are not evaluated or reported).

Calibration Status*	**Action**
CCC/SPCC pass criteria	calibration passes; all targets can be reported
CCC or SPCC fail	calibration fails; any targets that are from this sequence can be reported if the CCC criteria is met for that particular compound

If the initial calibration criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards.

10.2.8 After the initial calibration criteria (CCC/SPCC) have been met, each target is evaluated for linearity.

- If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

- If the %RSD of the target compound is greater than 15%, a calibration curve (linear, quadratic, etc) must be used for the quantitation of samples. A calibration curve may also be used for the compounds that have %RSD less than 15%

9 The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See Table 5 for a list of the compounds that are associated with the correct internal standard)
Cx = concentration of the compound being measured (ug/L)
Cis = concentration of the internal standard (50ug/L)

The initial calibration must be evaluated for the CCC and SPCC criteria even though the calibration curve may be used for quantitation.

The minimum number of calibration standards required for a regression curve are given in the following table:

TYPE OF CURVE	Minimum Number of Calibration Points
Linear (first order)	5
Quadratic (second order)	6
Polynomial (third)	7

Metals	Target Compound List[1]	Priority Pollutant List[2]	Appendix IX List[3]
Aluminum	X		
Antimony	X	X	X
Arsenic	X	X	X
Barium	X		X
Beryllium	X	X	X
Cadmium	X	X	X
Calcium	X		
Chromium	X	X	X
Cobalt	X		X
Copper	X	X	X
Iron	X		
Lead	X	X	X
Magnesium	X		
Manganese	X		
Mercury	X	X	X
Nickel	X	X	X
Potassium	X		
Selenium	X	X	X
Silver	X	X	X
Sodium	X		
Thallium	X	X	X
Tin			X
Vanadium	X		X
Zinc	X	X	X

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.1/ILM03.0/4.0

[2]Priority Pollutant: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX: 40 CFR 264, Appendix IX, 7/1/97

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	VG15	Modified 8015 Purge and Trap for Petroleum identification	VG15:07.15.98:5
	VG16	Volatile Petroleum Hydrocarbons (VPH)-Massachusetts Method	VG16:05.27.98:0
	VG25	The Analysis of Glycols by DAI-GC/FID	VG25:05.01.98:2
	VG30	THM Formation Potential	VG30:05.01.98:1
	VG40	The Analysis of Alcohols by DAI-GC/FID	VG40:05.01.98:1
	VG41	Direct Aqueous Injection (DAI) of Pulp and Paper Samples	VG41:08.19.98:0

Category	SOP Number	SOP Name	Document Control Number
'olatile Compounds by C	VG45	The Analysis of Industrial Solvents by DAI-GC/FID	VG45:05.01.98:1
Volatile Compounds by GC/MS	VM05	Volatile Compounds by GC/MS (EPA 624)	VM05:08.05.97:3
	VM06	Volatile Organic Compounds by Isotope Dilution GC/MS (Method 1624)	VM06:02.10.98:1
	VM15	Volatile Compounds in Drinking Water by GC/MS	VM15:05.01.98:1
	VM20	Volatile Compounds by GC/MS (8260)	VM20:07.15.98:3

SL SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC

Approval Signature: R. Wayne Robbins R. Wayne Robbins Title: Corporate QA Manager Date: July 17, 1998

ORGANOCHLORINE PESTICIDES AND PCBs BY GC
(Methods 608 and 8080/8081A/8082)

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls in liquid and solid extracts. Table 1 lists the routine target compounds and gives an example of the retention times of these target compounds. Example chromatograms are also included as an appendix to this SOP.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria are given in Section 5 of the current revisions of the SL quality assurance plans.

NOTE: The procedures for chlorinated pesticides (8081A) and PCBs (8082) have been separated in the latest update of SW-846. Until this update, the pesticides and PCBs were in SW-846 Methods 8080 and 8081. The extraction and the analysis are combined in this SOP 1) to reduce the time of extraction and analysis; and 2) to reduce the amount of solvent used in the procedures (one extraction instead of two). If interferences or high levels of non-PCB compounds are present, a portion of the extract will be subjected to the acid cleanup and reanalyzed. **Note that if the list of target analytes includes only a limited list of components (i.e. Toxaphene, Chlordane, or PCBs), these procedures may be abbreviated to address only the analytes of interest.**

2.0 SUMMARY OF METHOD

2.1 Aqueous and leachate samples are extracted with methylene chloride using a continuous liquid/liquid extractor (SOP EX30) or separatory funnel (SOP EX35). Soils are extracted with 1:1 hexane/acetone using a sonic dismembrator (SOP EX40). The solvent is evaporated, the residue exchanged into hexane, and the sample adjusted to a final volume of 10 mL. This method may also incorporate Florisil, copper, acid, and gel permeation chromatography (GPC) cleanup. Analysis of the extract is performed on a GC equipped with dual capillary columns connected to dual electron capture (EC) detectors. Detection and confirmation are performed simultaneously when using the dual column/dual detector configuration.

2.2 The procedures for waste dilution are given in SL SOP EX42. SL SOP SG60 contains the acid and permanganate cleanups for PCBs and includes a procedure for the elimination of sulfur with copper.

2.3 This method is based on the guidance in SW-846 Methods 8000B, 8080, 8081A, and 8082 and 40 CFR 136 Method 608.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially hazardous situations.

3.2 The analyst should wear an apron or lab coat, gloves, and eye protection when handling extracts. Dilutions should be performed under a hood or in a well-ventilated area.

3.3 The analyst must be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in the analysis of pesticides and PCBs. Many of these compounds are suspected carcinogens.

4.0 INTERFERENCES

4.1 Glassware should be scrupulously cleaned and solvent-rinsed in accordance with SL SOP AN60 to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination form the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The following table lists cleanups may be implemented to eliminate or reduce interferences in order to achieve the lowest possible detection limits. If matrix interferences continue after a clean up has been performed, the sample is diluted as needed for data analysis. If a cleanup is used, the method blank must also be subjected to the cleanup.

CLEAN-UP PROCEDURE	SL SOP	APPLICATION	EFFECTIVENESS
Florisil	EX62	pest/PCBs	Eliminates polar non-target compounds
Sulfuric acid / permanganate	EX60	only PCBs	Eliminates most non-target interferences
Copper	EX60	pest/PCBs	Eliminates elemental sulfur
GPC	EX61	pest/PCBs	Eliminates high molecular weight non-target compounds

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

Aqueous samples are collected in 1-L glass containers with Teflon-lined caps. Soil/sediment samples are collected in wide mouth glass jars equipped with Teflon lined caps. No preservative is added. The samples must be stored at 4°C until extraction. Extraction must be performed within 7 days for aqueous samples and within 14 days of sampling for soils/solids. The extracts must be stored at 4°C and must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperatures programmable, equipped with single or dual electron capture (EC) detectors and a compatible autosampler. The GC should be capable of housing two columns and have make-up gas lines for each detector

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended

DB-5 fused silica capillary column 30 M x 0.53 mm ID x 1.5 um film (J&W or equivalent)
DB-608 fused silica capillary column 30 M x 0.53 mm ID x 0.83 um film (J&W or equivalent)
<Or>
DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-17 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
<Or>
DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-1701 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)

6.4 Microsyringes, 10-, 25-, 50-, 100-, 250, 500-uL; Hamilton or equivalent

6.5 Volumetric flasks, 10-, 50-, 100-, 200-mL Class A

6.6 Autosampler vials, septa, and caps; compatible with the autosampler

7.0 REAGENTS

Hexane- pesticide grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in SL SOP AN43:*Standard Preparation*.

The lab should purchase certified solutions from SL-approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SL SOP AN43 for guidance for standard preparation.

8.1 Calibration Standard Recipes

The recipes given in Appendix A are provided for guidance. If the recipes used are different from those given in Table 2, the lab must clearly document the recipe as a controlled posting or as a narrative in the traceability log. Note that the preparation of some of the calibration standard will include intermediate level standards.

The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the SL QAPs.

9.0 SAMPLE PREPARATION

The sample preparation procedures are described in the following SL SOPs:

Procedure	Matrix	SL SOP
Continuous Liquid-liquid extraction	aqueous and leachates	EX30
Separatory funnel extraction	aqueous and leachates	EX35
Ultrasonic extraction	soils and sediments	EX40
Waste dilution	waste samples (oils, products, etc)	EX42
Zymark extract concentration	aqueous, leachate, and soil extracts	EX50

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions

The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations may be used for the analysis of pesticides and PCBs. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column or a glass tee to provide simultaneous detection and confirmation of the target analytes. The following stationary phase pairs are recommended:

DB-5 fused silica capillary column 30 M x 0.53 mm ID x 1.5 um film (J&W or equivalent)
DB-608 fused silica capillary column 30 M x 0.53 mm ID x 0.83 um film (J&W or equivalent)
<Or>
DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-17 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
<Or>
DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-1701 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
<And>
Guard Column 6 m x 0.53 mm ID

A guard column is recommended to help protect the analytical columns. Capillary columns (0.32mmID) generally do not have the capacity of the megabore columns and should not be used for analysis of oily samples.

10.1.2 GC Parameters

Injector: 240 – 260°C
Detector: 300°C
Carrier Gas Flow: Helium at 5 mL/min (per column)
Make-up Gas Flow: Nitrogen at 25 mL/min (per detector)-see manufacturer's recommended flows

Temperature program (example chromatogram temperature program):

Initial Temp:	160 C
Initial Hold:	4.0 min
Program Rate:	10 C/min
Final Temp:	270 C (hold for 10 minutes)
Injected Volume:	2-4uL - 1-2uL per column (single injection into guard column and "Y" splitter)

NOTE: These conditions and parameters are given for guidance. The conditions and parameters may be modified to optimize the analytical system.

10.2 Calibration
The external standard calibration technique is employed for the determination of the concentration of pesticides and PCBs

10.2.1 Column Evaluation
The column(s) must be evaluated prior to the analysis of the calibration standards. The column evaluation is performed by injecting Endrin and p,p'-DDT and calculating the percent breakdown of these compounds.. The column evaluation does not have to be performed if PCBs only are the target compounds. PCBs are stable and not subject to breakdown in the injection port.

10.2.1.1 If the instrument has not been in use for greater than one day, a "priming" analysis may be beneficial. The analysis of a relatively high concentration pesticide or PCB standard may help to stabilize the response of the very sensitive EC detector. Inject a standard that is about 10x the concentration of the highest calibration standard and allow the instrument to cycle through the temperature program. It is not necessary to acquire the data but the baseline should be monitored before and after the priming analysis to gauge the condition of the detector. A hexane blank should be analyzed after the analysis of the priming standard and before the %breakdown check.

NOTE: The "priming" standard should be injected by hand to avoid contaminating the autosampler syringe.

Inject the Endrin/DDT breakdown standard. Check the peak integrations and calculate the breakdown as follows:

$$\%\text{Breakdown Endrin} = \frac{\text{Area (Endrin ketone)} + \text{Area (Endrin aldehyde)}}{\text{Area (Endrin)} + \text{Area (Endrin ketone)} + \text{Area (Endrin aldehyde)}} \times 100$$

$$\%\text{Breakdown DDT} = \frac{\text{Area (DDE)} + \text{Area (DDD)}}{\text{Area (DDT)} + \text{Area (DDE)} + \text{Area (DDD)}} \times 100$$

The breakdown for each compound must be less than 15%. If the breakdown exceeds 15%, the instrument will require column and/or injector port maintenance. The maintenance may include but is not limited to replacing the septum, clipping the front of the column, and replacing the glass injector sleeve.

10.2.2 Initial Calibration

The initial calibration for this procedure involves the analysis of a multi-point calibration for the single peak pesticides, a multi-point curve for a mixture of AR1016 and AR1260, and single point calibration standards for the remaining Aroclors, Toxaphene, and technical Chlordane. Note that depending on instrument resolving power, the initial calibration of single components may be achieved using one mix containing all single components, or using 2 mixes with the target list split between the mixes.

Note that the following offers two (2) options for quantitation – calibration curve or average RF. Only one need be chosen per analyte.

10.2.2.1 Prepare and inject the calibration standards using the guidelines listed in Section 8 of this SOP. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in Section 10.3.

10.2.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?

>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?

>Are the peaks symmetrical? Is there tailing or fronting?

>Are the areas of the peaks normal for the sensitivity setting being used?

10.2.2.3 Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.2.4 **Calibration Curve Option:** A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPs for other exceptions to using non-linear curve fitting.

10.2.2.5 **Average RF/CF Option**:

Calculate the response factor of each calibration standard, the average response factor and the relative standard deviation using the following equations:

Response Factor

$$X_{RF} = \frac{mg/mL}{area} = \frac{mg}{mL\ area}$$

Note that the inverse relationship can also be used to evaluate the response of the detector. Dividing the area of the standard by the concentration is referred to as the calibration factor.

Average Response Factor

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 + \ldots RFn}{n}$$

Relative Standard Deviation

$$\%RSD = \frac{standard\ deviation}{\overline{RF}} \otimes 100$$

and

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}}$$

Where

RF_i = response factor of the individual calibration level
$\overline{RF}$ = average response factor

If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear and the average response factor (or calibration factor) may be used for quantitation.

NOTE ON 8000-SERIES METHODS: If the %RSD for any target compound in the initial calibration exceeds 20%, a calibration curve must be constructed and evaluated for that compound. In addition to meeting the correlation coefficient requirements in 10.2.1.3, the *average %RSD for all of the compounds in the initial calibration* ***must*** *be less than or equal to 20% for the initial calibration to be used.*

10.2.3 Calibration is verified every 12 hours or 20 samples, whichever is more frequent by the analysis of continuing calibration check standards. The single component pesticides are verified using the mid-level single component pesticide standard(s). PCB calibration is verified using the mid-point AR1660 standard. Additionally, breakdown must be evaluated every 12 hours. Refer to section 10.2.1 for procedures for evaluating breakdown. Toxaphene and technical chlordane are only verified when encountered in samples or when they are the only analytes of interest. Note that sections 11.1.5.1 – 11.1.5.3 detail specific calibration procedures for the multiresponse analytes. Note that the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive for the compound(s) failing high are detected, these samples may be reported.

10.2.3.1 Analyze the standards. Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Response Factor:

$$X_{RF} = \frac{mg/mL}{area} = \frac{mg}{mL \quad area}$$

Note that the inverse relationship can also be used to evaluate the response of the detector. Dividing the area of the standard by the concentration is referred to as the calibration factor.

Calculate the percent drift or percent difference between the initial and continuing calibration:

$$\%Drift = \frac{result - expected}{expected} x\ 100$$

Where
result = concentration or nanograms on-column of the calibration check standard quanted against the curve
expected = true concentration or nanograms on-column of the calibration check standard

$$\%Difference = \frac{RF_{init} - RF_{cont}}{RF_{init}} x\ 100$$

Where
RF_{init} = average response factor from the initial calibration curve
Rf_{cont}= response factor from the continuing calibration standard

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If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor is used for quantitation.

If the percent difference is greater than 15%, another continuing calibration standard should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the continuing calibration criteria are not met, a new initial calibration must be prepared and analyzed.

NOTE ON 8000-SERIES METHODS: If the % difference or % drift for any target compound in the CCV exceeds 15%, the calibration curve can still be used for quantitation if the average % difference or % drift for all of the compounds in the CCV is less than or equal to 15%.

10.3 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the respective summaries included after Section 15 of this SOP.

10.3.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks (hexane) may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.3.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. For the single point multicomponent products, the extract should be diluted until the area is no more than a factor of two above the area of the single point standard (see section 11.1 for quantitation of multi-peak target compounds).

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used (*F34 or *F42) or qualification in a case narrative provided to the client. For TCLP analyses, every effort should be made to achieve the regulatory level without substantial instrument overload.

For clients who demand we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve (i.e., a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client). Project managers and lab staff must work together to balance client satisfaction with productivity.

10.4 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SL SOP AN66: Determination of Retention Time Windows for Gas Chromatographic Analyses.

11.0 DATA ANALYSIS/CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times), the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration, and the integration of the peaks. Manual integration must be documented in accordance with SL SOP AN65. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data.

The judgement and experience of the analyst and his/her colleagues is an important part of the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks correctly integrated?
- Are there co-eluting peaks or matrix interferences?

11.1 Qualitative analysis

Identification of the target compounds is based on retention time. The analyst should scan the sample chromatogram for the target compounds on the primary analytical column. The analyst should use the retention time (RT) window calculated around the CCV as guidance for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds to help gauge possible shifts in the RT of the target compounds. See SL SOP AN66.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peaks areas that exceed the established working range of the calibration curve may result in significant retention time shifts; therefore, all peaks which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (10.3.2).

NOTE: Elemental sulfur present in the extract will cause a large, irregular interfering peak in the middle of the chromatogram. When sulfur interference is suspected samples should be copper cleaned as detailed in SL SOP EX60.

11.1.1 The surrogates should be evaluated first to check for shifts in retention times and to evaluate the surrogate recovery.

Two surrogates are spiked: 2,3,4,6-tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB). DCB should be evaluated as the main surrogate and TCMX is evaluated if there is matrix interference with DCB. The recovery criteria are given in Section 5 of the SL QAP.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Generally, it is expedient to calculate the recovery of both surrogates on one chromatogram. If these recoveries are deemed acceptable, the other channel is not evaluated for recovery. If interferences are suspected, evaluate recoveries on the other channel. Note that given this nature, an extract is considered acceptable if one of the 4 potentially calculated recoveries is acceptable.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to section 13 of the current QA Plan regarding this issue.

11.1.2 Label and calculate the concentration of each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, the presence of multi-response peaks (technical chlordane, Toxaphene, and the Aroclors) and the overall shapes of the peaks.

If the concentration is below the detection limit, the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. A line should be drawn through any data system generated hits not called by the analyst. Peaks over-range are handled using dilutions as detailed above (10.3.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds to help gauge possible shifts in the RT of the target compounds.

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

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If the relative percent difference is less than 40%, the presence of the target compound is confirmed and the lower concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A large relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. If interference is detected, flag the result to note the disparity between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution or perform additional cleanup as detailed in SOP EX60, EX61, EX62 is appropriate.

The following table summarizes the general guidance for the evaluating of chromatographic data. The table assumes that the calibration criteria have been met and that the sample has acceptable associated surrogate and lab spike recoveries.

PEAK INFORMATION	ACTION	REPORT*
No peaks found on primary or secondary column		Report < RL
Peak found within RTW on primary column	Peak is tentatively identified as the target	If concentration < RL, report < RL If concentration > RL, evaluate confirmation column.
Peak found within RTW on confirmation column	Peak is confirmed as the target	If concentration < RL, report <RL If concentration >RL, calculate %RPD -if %RPD <40%, report lower concentration of primary and confirmation analyses. -if %RPD >40%, flag result to note the disparity Case narrative or note to PM may be required for complex matrices.

*RL may be the SL Reporting Limit in Table 5 of the CQAP or may be defined by the client QAP or contract.

The analyst must clearly show how the reported sample results were determined. False hits identified as targets by the data system should be "struck out" by drawing a line through the hit on the quantitation report. Manual calculations and integrations must be documented.

11.1.4 Identification "Tools"

11.1.4.1 Relative Retention Time

The retention time of a surrogate compound provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions.. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification.

11.1.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

11.1.5 Qualitative Analysis of Multiple Peak Compounds

Identification of multicomponent pesticides/PCBs is based on the recognition of their chromatographic patterns. Quantitation is performed using the area of characteristic peaks in the sample and standard using external calibration procedures.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be warranted. Suggested cleanup options are given in Section 4.

11.1.5.1 PCBs

PCBs are generally reported as Aroclors. The Aroclors have varying levels of PCB congeners with the last two numbers in the Aroclor designation indicating the weight percent of chlorine. For example, AR1221 is 21% chlorine by weight; AR1260 is 60% chlorine by weight. The 12- in the Aroclor designation represents the biphenyl molecule. The exception to this naming convention is AR1016, which is about 42% chlorine by weight. (Note that AR1026 and AR1242 have similar chromatograms - both Aroclors have almost the same weight of chlorine by weight and nearly the same PCB congeners.)

Aroclors are identified by matching the pattern of the sample with standards analyzed under the same analytical conditions. Interference may occur due to the presence of non-target analytes or due to "weathering" of the Aroclor in the environment. The presence of multiple Aroclors will also complicate the identification and quantitation of the Aroclors. Many matrix interferences may be reduced or eliminated by treating the sample extract with copper, sulfuric acid, and permanganate prior to analysis. SL SOP EX60 details this procedure. *NOTE: Do not use the acid or permanganate cleanup on the entire extract if pesticides are also to be reported as many of the pesticides are not stable in acid or strong oxidizer.*

When a pattern matching an Aroclor is encountered, it may be quantitated using either the 3-5 characteristic peaks (preferred) or total area response or by using 3-5 characteristic peaks (preferred). Total area quantitation should only be used as detailed below. Residues of either AR1016 or AR1260 are quantitated using the average RF/CF determined during initial calibration. The other Aroclors are quantitated against the RF/CF determined from their single-point analysis during initial calibration. Samples should be diluted when the amount of PCB in a sample extract exceeds the calibration range defined in initial calibration. Note that the AR1660 standard defines the working range for all the Aroclors (i.e. if AR1660 was calibrated from 1 to 100 ug/ml, and a sample extract was analyzed containing 200 ug/ml of AR1232, that extract would require dilution to get the amount of AR1232 to be less than 100 ug/ml.)

In the 3-5 peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of PCB in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 3-5 resulting concentrations are averaged to provide the final result for the sample.

"Weathering" is the loss of part of the Aroclor pattern due to biological or chemical degradation of individual PCBs. When weathering is suspected, try to match the later eluting peaks first. Flag the results for a weathered Aroclor pattern as tentatively identified and make a note in the case narrative if provided.

The presence of multiple Aroclors can be a problem to identify since most Aroclors have at least a few PCBs in common. The easiest case would be to have early and late eluting Aroclors present. The most difficult cases will involve the presence of Aroclors with the same relative chlorine level. In cases where the identification of Aroclors cannot be clearly determined, quantify the Aroclors against the Aroclor that most closely matches the pattern of the sample using the total area of all peaks within the pattern range. The result should be flagged and noted in the case narrative if provided.

NOTE: When choosing individual peaks for quantitation, compare their responses in the sample and standard. If the peaks chosen do not correlate well (i.e. ratios to other peaks are close) between the sample and standard, review the chromatograms for other possible peaks for quantitation.

11.1.5.2 Toxaphene

Toxaphene is a mixture of chlorinated camphenes, which has a complex and characteristic pattern when analyzed by GC-ECD. A single Toxaphene standard is analyzed during the initial calibration for the purpose of pattern identification in samples. When a Toxaphene residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for Toxaphene should be analyzed. Alternatively, single points may be prepared with Toxaphene concentrations within 2x the Toxaphene quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the Toxaphene standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of Toxaphene-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours.

If the sample and standard chromatogram agree well, Toxaphene is quantitated using 5 characteristic peaks (similar to the PCB approach, above). If the sample and standard pattern do not agree as well (i.e. individual peak ratios do not agree as well, but all the major Toxaphene components in the standard are present in the sample), a total area integration is more appropriate. To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene components in the standard. Note that 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 and that extraneous peaks or humps (contributed by non-Toxaphene components) are NOT included in the quantitation.

When Toxaphene is determined using the 5 peak approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionally larger or smaller in the sample compared to the standard.

In the 5-peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 5 resulting concentrations are averaged to provide the final result for the sample.

11.1.5.3 Chlordane – 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. The following components are significant: α and γ Chlordane, trans-Nonachlor, Heptachlor, and Heptachlor-epoxide. The α and γ Chlordane isomers are the most prevalent and their detection as single components is a good indicator that Technical Chlordane may be present.

The following sections discuss three specific options: reporting Technical Chlordane, reporting Chlordane (not otherwise specified-"NOS"), and reporting the individual Chlordane components that can be identified under their individual CAS numbers.

When the GC pattern of the residue resembles that of the Technical Chlordane standard, quantitate Chlordane residues by comparing the area of 3 to 5 major peaks. Heptachlor and heptachlor epoxide should not be included in this quantitation but rather should be quantitated and reported separately.

The GC pattern of a Chlordane residue in a sample may differ considerably from that of the Technical Chlordane standard. In such instances, it may not be practical to relate a sample chromatogram back to the lab's Technical Chlordane Standard. Therefore, depending on the objectives of the analysis, the analyst may choose to report the sum of all the identifiable Chlordane components as "Chlordane (n.o.s.)" under the CAS number 57-74-9. This option should only be used at the direction of or in consultation with a project manager.

The third option is to quantitate the peaks of a-Chlordane and y-Chlordane separately against the appropriate reference materials, and report these individual components.. This option should be used only after consultation with the project manager. Note that quantitation values determined from the individual chlordane isomers will be substantially lower than those reported for the technical product.

When a Technical Chlordane residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for technical chlordane should be analyzed. Alternatively, single points may be prepared with technical chlordane concentrations within 2x the Technical Chlordane quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the technical chlordane standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of technical chlordane-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours. **Note that these procedures are not necessary if the lab is reporting chlordane as the α and γ chlordane isomers, not as the technical product.**

11.2 Calculations

Aqueous/Liquid Samples

If the calibration (regression) curve option is used, the sample concentration is calculated:

$$Concentration(ug/L) = Ccurve \otimes QF$$

where
Ccurve = concentration of analyte from curve (ug/mL)
QF = "quant factor"

This equation expands to:

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{V}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

If the response factor option is chosen for quantitation:

$$Concentration(ug/L) = RF \ x \ response \ x \ QF$$

where
RF = response factor
QF = "quant factor"
esponse = area or height of target compound

This equation expands to:

$$Concentration(ug/L) = \frac{ug/mL}{response} \otimes response \otimes \frac{F \otimes DF}{V}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

Soils/Solids Samples

If the calibration curve option is used, the sample concentration is calculated:

$$Concentration(ug/kg, dw) = Ccurve \otimes QF$$

where
Ccurve = concentration of analyte from curve (ug/mL)
QF = "quant factor"

This equation expands to:

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
W = weight of sample extracted (kg)
solids = (percent solids)/100

If the response factor option is chosen for quantitation:

$$Concentration(ug/kg, dw) = RF \otimes response \otimes QF$$

where
RF = response factor
QF = "quant factor"
response = area or height of target compound

This equation expands to:

$$Concentration(ug/kg, dw) = \frac{ug/mL}{response} \otimes respnse \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)
W = weight of sample extracted (kg)
solids = (percent solids)/100

Accuracy is expressed as percent recovery of the target analyte. The theoretical concentration, Ct, of the spike is calculated:

LIQUIDS

$$Ct(ug/L) = \frac{Cs \otimes Vs}{Vsample}$$

where
Cs = concentration of the spiking solution, ug/mL
Vs = volume of spiking solution added to the sample, mL
Vsample = volume of sample spiked, L

SOILS

$$Ct(ug/kg, dw) = \frac{Cs \otimes Vs}{Wsample \otimes solids}$$

where
Cs = concentration of the spiking solution, ug/mL
Vs = volume of spiking solution added to the sample, mL
Wsample = weight of sample spiked, kg
solids = (percent solids)/100

The percent recovery is calculated:

$$\%REC = \frac{(Cms - Csample)}{Ct} \otimes 100$$

where
Cms = concentration determined from the analysis of the spiked sample (MS or LCS)
Csample = concentration of the unspiked sample (for LCS, let Csample = 0)
Ct = theoretical concentration of the MS or LCS

If the concentration of the unspiked sample is less than the RL, let Csample = 0.

Precision is measured as the relative percent differnce (%RPD), which is calculated as :

$$\%RPD = \left| \frac{(\%REC1 - \%REC2)}{\left(\frac{\%REC1 + \%REC2}{2}\right)} \right| \otimes 100$$

where
%REC1 = percent recovery of the MS or LCS
%REC2 = percent recovery of the MSD or LCSD

12. 0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The **analytical batch** is discussed in SL SOP AN02: *Analytical Batching*, and these criteria are summarized in the SOP Summary included in this SOP.

12.2 The **method detection limit (MDL)** is defined as the concentration of the an analyte that can be measured with a 99% confidence that the result is greater than zero. See SL SOP CA90: *Procedure for Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items are included in this revision. See Section 10 of the current SL QAP.

14.0 TROUBLESHOOTING

No items are included in this revision.

15.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

2. *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office: Washington, DC, July 1, 1988.

3. Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*, current revision.

Appendix A:CHLORINATED PESTICIDES AND PCBs STANDARDS PREPARATION

Intermediate Surrogate Standard

STOCK STANDARD	CONC. (ug/mL)	uL of stock	fvol (mL)	conc (ug/mL)
Pesticide Surrogate	200	25	1.0	5.0

Individual Standard Mix A

STOCK STANDARD	CONC. (ug/mL)	ISMA-1 *	ISMA-2 *	ISMA-3 *	ISMA-4 *	ISMA-5 *
Pesticide Std A	5-100	10uL	20uL	40uL	60uL	100uL
Pesticide Surrogate Intermediate	5.0	10uL	20uL	40uL	60uL	100uL

*microliters to 10mL hexane(final volume)

Individual Standard Mix B

STOCK STANDARD	CONC. (ug/mL)	ISMB-1 *	ISMB-2 *	ISMB-3 *	ISMB-4 *	ISMB-5 *
Pesticide Std B	5-50	10uL	20uL	40uL	60uL	100uL
Pesticide Surrogate Intermediate	5.0	10uL	20uL	40uL	60uL	100uL

*microliters to 10mL hexane(final volume)

Concentrations of the Target Compounds in the Calibration Standards

Target compound	ISMA-1 (ug/mL)	ISMA-2 (ug/mL)	ISMA-3 (ug/mL)	ISMA-4 (ug/mL)	ISMA-5 (ug/mL)
g-BHC(Lindane), TCMX, DCB	0.0050	0.010	0.020	0.030	0.050
Aldrin, Dieldrin, Endosulfan I, Heptachlor, Heptachlor epoxide	0.010	0.020	0.040	0.060	0.10
p,p'-DDT, Endosulfan II	0.020	0.040	0.080	0.12	0.20
Endrin aldehyde	0.0025	0.050	0.10	0.15	0.25
Methoxychlor	0.10	0.20	0.40	0.60	1.0

Appendix A

Target compound	ISMB-1 (ug/mL)	ISMB-2 (ug/mL)	ISMB-3 (ug/mL)	ISMB-4 (ug/mL)	ISMB-5 (ug/mL)
a-BHC, TCMX, DCB	0.0050	0.010	0.020	0.030	0.050
Aldrin, b-BHC, d-BHC, a-Chlordane, g-Chlordane, p,p'-DDE, Endrin	0.010	0.020	0.040	0.060	0.10
p,p'-DDD, Endosulfan sulfate, Endrin ketone	0.020	0.040	0.080	0.12	0.20

CHLORINATED PESTICIDES STANDARDS PREPARATION

Technical Chlordane Intermediate Standard

STOCK STANDARD	CONC. (ug/mL)	uL of stock	fvol (mL)	conc (ug/mL)
Technical Chlordane	1000	100	1.0	100

Toxaphene Single Intermediate Standard

STOCK STANDARD	CONC. (ug/mL)	uL of stock	fvol (mL)	conc (ug/mL)
Toxaphene	1000	100	1.0	100

Single point calibration standards are analyzed for t-Chlordane (0.20ug/mL) and Toxaphene (1.0ug/mL) with the routine analytical sequence. If either of these target compounds is detected in field samples, a three point curve (EPA608) or a five point curve (EPA 8080/8081) must be analyzed and evaluated and the sample extracts containing these targets re-analyzed.

Technical Chlordane Five-point Curve- 0.10, 0.20, 0.40, 0.60, 0.80ug/mL

STOCK STANDARD	CONC. (ug/mL)	TCHLOR -1*	TCHLOR -2*	TCHLOR -3*	TCHLOR -4*	TCHLOR -5*
Technical Chlordane	100	10	20	40	60	80

*microliters to 10mL hexane(final volume)

Toxaphene Five-point Curve- 0.10, 0.25, 0.50, 1.0, 2.0ug/mL

STOCK STANDARD	CONC. (ug/mL)	TOX -1*	TOX -2*	TOX -3*	TOX -4*	TOX -5*
Toxaphene Intermediate	100	10	25	50	100	200

*microliters to 10mL hexane(final volume)

Appendix A

AROCLOR STANDARDS PREPARATION

The Aroclor (PCB) standards can be used for EPA Method 8080/8081Ato report the routine list of Aroclors. The standards are prepared in hexane at a final volume of 10mL and stored at 4C. AR1660 is used to establish calibration linearity for the Aroclor mixtures and is used as the calibration check. Use AR1660 as a guide if calibration curve of other Aroclors required.

Intermediate AR1660/surrogate standard

STOCK STANDARD	CONC (ug/mL)	uL of stock to 10mL hexane	conc. of intermediate std(ug/mL)
Ar1016	1000	100	10
Ar1260	1000	100	10
TMX/DCB	200	10	0.2

Working Level AR1660/surrogate standards

INTERM STANDARD	CONC (ug/mL)	AR1660 -1*	AR1660 -2*	AR1660 -3*	AR1660 -4*	AR1660 -5*
AR1660/ Surrogate	10 0.2	100 (0.10)	200 (0.20)	500 (0.50)	1000 (1.0)	2000 (2.0)

*microliters (mL) of intermediate standard used to prepare 10mL of working standard in hexane

Concentrations of the AR1660 Standards

Calibration Std	AR1016(ug/mL)	AR1260(ug/mL)	surrogates
AR1660-1	0.10	0.10	0.002
AR1660-2	0.20	0.20	0.004
AR1660-3	0.50	0.50	0.010
AR1660-4	1.0	1.0	0.020
AR1660-5	2.0	2.0	0.040

AR2154 Calibration Standard

STOCK STANDARD	CONC (ug/mL)	uL of stock to 10mL hexane	conc. AR2154-1 (ug/mL)
Ar1221	1000	10	1.0
Ar1254	1000	10	1.0

AR1232 Calibration Standard

STOCK STANDARD	CONC (ug/mL)	uL of stock to 10mL hexane	conc. AR1232-1 (ug/mL)
Ar1232	1000	10	1.0

Appendix A

AR1242 Calibration Standard

STOCK STANDARD	CONC (ug/mL)	uL of stock to 10mL hexane	conc. AR1242-1 (ug/mL)
Ar1242	1000	10	1.0

AR1248 Calibration Standard

STOCK STANDARD	CONC (ug/mL)	uL of stock to 10mL hexane	conc.AR1248-1 std(ug/mL)
Ar1248	1000	10	1.0

DDT/Endrin Breakdown Evaluation Standard

STOCK STANDARD	CONC (ug/mL)	uL of stock to 10mL hexane	conc.AR1248-1 std(ug/mL)
Column Evaluation	2	10	0.020

The evaluation standard also contains aldrin and dibutyl chlorendate.

Appendix B

608/8080/8081/8082 METHOD SUMMARY

HOLD TIMES

MATRIX	Preservative/ storage*	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days	40 days

*Storage temperature is 4C with a control criteria of less than 6C with no frozen samples

EXTRACTION

Aqueous: Approximately 1L of sample (contents of container) using continuous or separatory funnel extraction at pH 5-9 with methylene chloride; exchange to hexane and concentrate to final volume of 10mL

Soil/Solids: Approximately 30g of sample using sonication with 1:1 acetone/hexane;
Concentrate to final volume of 10mL in hexane

Wastes: Approximately 1g of sample diluted to final volume of 10mL with hexane

ANALYSIS

Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external standard calibration

SEQUENCE-600-series

Endrin/p,p'-DDT breakdown evaluation (recommended)
Initial Calibration- 3 point single peaks compounds 1 point all Aroclors 1 point toxaphene 1 point technical chlordane
Initial Calibration Verification (ICV)
Sample analyses
Continuing calibration verification (CCV)-daily-every 24 hours Single peak compounds AR1660
RL standard(optional; required by state or client QAP)
Sample analyses

Sequence continues until all samples have been analyzed or the CCV fails the acceptance criteria. If a multi-peak target compound is detected, the extract is reanalyzed with a 3-point curve.

SURROGATE(S):

Tetrachloro-m-xylene- 0.20ug/L
Decachlorobiphenyl-0.20ug/L

BATCH QC

Method blank
LCS/LCSD- full target of single peak analytes @ 0.20ug/L
MS/MSD- full target of single peak analytes @ 0.20ug/L

Appendix B

SEQUENCE-8000-series

STANDARD/SAMPLES
Endrin/p,p'-DDT breakdown (every 12 hours)
Initial Calibration- 5 point single peaks 5 point Ar1660(note1) 1 point Toxaphene 1 point tech Chlordane 1 point remaining Aroclors
Up to twenty sample extracts
Endrin/p,p'-DDT breakdown (every 12 hours)
Continuing calibration check midpoint single peaks midpoint Ar1660(note1)
RL Standard(optional)-lowest point on the calibration curve if required by state or client QAP
Up to twenty sample extracts

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All samples extract analyses must be bracketed by acceptable verification standards.

Note 1-A mixture of Ar1016 and Ar1260 will be used to calibrate and verify the response for PCBs and to verify the response for Toxaphene and technical Chlordane in the continuing calibration.

SURROGATE (S):
Tetrachloro-m-xylene- 0.20ug/L
Tecachlorobiphenyl-0.20ug/L

BATCH QC
Method blank
LCS- CQAP subset
MS/MSD- CQAP subset

Parameter	Aqueous(ug/L)	Soils(ug/kg)
Lindane	0.20	6.0
Aldrin	0.20	6.0
Heptachlor	0.20	6.0
Dieldrin	0.50	15
Endrin	0.50	15
p.p'-DDT	0.50	15

Appendix B

8000-SERIES CALIBRATION

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The curve is inspected before proceeding with sample analysis. The correlation coefficient must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD of the calibration curve is less than or equal to the acceptance criteria, the average response factor can be used for quantitation. A midpoint calibration verification standard must be analyzed at the frequency specified in the method SOP as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

8000-series ICAL exception if the %RSD criteria is used to evaluate the calibration curve:
If one or more compounds fail the %RSD criteria, the calibration curve can be used for quantitation if the average %RSD of ALL of the compounds in the calibration standard is less than or equal to 20% and the user of the data is notified of the compounds that fail.

8000-series CCAL exception for %drift or %difference criteria:
If one or more compounds fail the %drift or %difference criteria, the calibration may still be considered valid if the average %drift or %difference of ALL of the compounds in the calibration standard is less than or equal to 15% and the user of the data is notified of the compounds that fail.

If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Appendix B

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
p,p'-DDT and Endrin breakdown check -required for 8000-series, recommended for 600-series	Initially and every 12 hours	% breakdown of both compounds less than 20%	-re-analyze check solution -perform injector port and/or column maintenance and re-analyze
Initial Calibration- 600-series:3 point minimum with lowest point at RL 8000-series:5 point minimum with lowest point at RL	Initially prior to sample analysis, when major instrument maintenance performed, or when CCV fails	**600-series:** 1) RSD of each target <= 10%; OR 2) plot regression curve CC>=0.99 for each target **8000-series:** 1) RSD of each target <= 20%; OR 2) plot regression curve CC>=0.99 for each target (see previous page for exceptions)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification(CCV)	After every ten to twenty sample analyses and at the end of the sequence if external standard calibration is used	Percent difference or drift <= 15% (see previous page for exceptions)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in Section 5 of the SL CQAP	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP

Appendix B

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Lab control sample (LCS)- subset of targets in SL CQAP	Per batch (If MS/MSD cannot be performed , the LCS must be performed in duplicate)	Recoveries within SL CQAP Section 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP
Matrix spike(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within SL CQAP Section 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP
Surrogates	All samples, method blanks, and QC	Recoveries within SL CQAP Table 5 limits See section 11.1.1. for specifics	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP
Reporting limit(RL) standard-standard at the reporting limit used to verify sensitivity of the instrument	Daily(optional-see specific state or client requirements for frequency)	Detected with reasonable sensitivity	-Reanalyze RL standard -Remake and reanalyze RL standard -Perform instrument or column maintenance, recalibrate, and reanalyze associated samples
Retention time window determination	See guidance in SL SOP AN66	See guidance in SL SOP AN66	Use guidance in SL SOP AN66: *Determination of Retention Time Windows in Gas Chromatographic Analyses*

Appendix B

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Initial demonstration of Capability (the analyst has to perform the IDOC for either of the analogous 600 or 8000 series methods- not both)	Per analyst	Within the 600- or 8000-series method limits	-Reanalyze QC sample for the targets that failed to meet the criteria
Method detection limit(MDL)	See SL SOP CA90	Evaluate data using criteria SL SOP CA90	-Evaluate data. Check calculations. -Reanalyze MDL samples.

Appendix B

TABLE 1			
Compound	RT COL 1	RT COL 2	CHROMAT
Tetrachloro-m-xylene	5.26	6.65	
Alpha-BHC	7.39	7.71	
Gamma-BHC (Lindane)	8.63	8.65	
Beta-BHC	8.80	8.39	
Heptachlor	9.54	10.61	
Delta-BHC	9.83	9.19	
Aldrin	10.38	11.53	
Heptachlor epoxide	11.96	12.51	
Gamma-Chlordane	12.31	13.04	
Alpha-Chlordane	12.72	13.41	
Endosulfan I	12.86	13.42	
4,4'-DDE	13.39	13.84	
Dieldrin	13.63	14.05	
Endrin	14.45	14.53	
4,4'-DDD	14.75	14.83	
Endosulfan I	14.97	14.78	
4,4'-DDT	15.54	15.78	
Endrin aldehyde	15.76	15.21	
Endosulfan sulfate	15.98	15.71	
Dibutyl chlorendate	16.73	17.80	
Methoxylchlor	17.60	17.04	
Endrin ketone	17.79	16.82	
Decachlorobiphenyl	21.36	22.36	
Technical chlordane	MR	MR	
Toxaphene	MR	MR	
AR 1221	MR	MR	
AR1232	MR	MR	
AR1016	MR	MR	
AR1242	MR	MR	
AR1248	MR	MR	
AR1254	MR	MR	
AR1260	MR	MR	
Isodrin	11.85	12.45	
Chlorobenzilate	14.51	14.79	
AR1268	MR	MR	

MR = multi-peak/multi-response compounds

COL1 = DB608 COL2 = DB-5

Appendix C

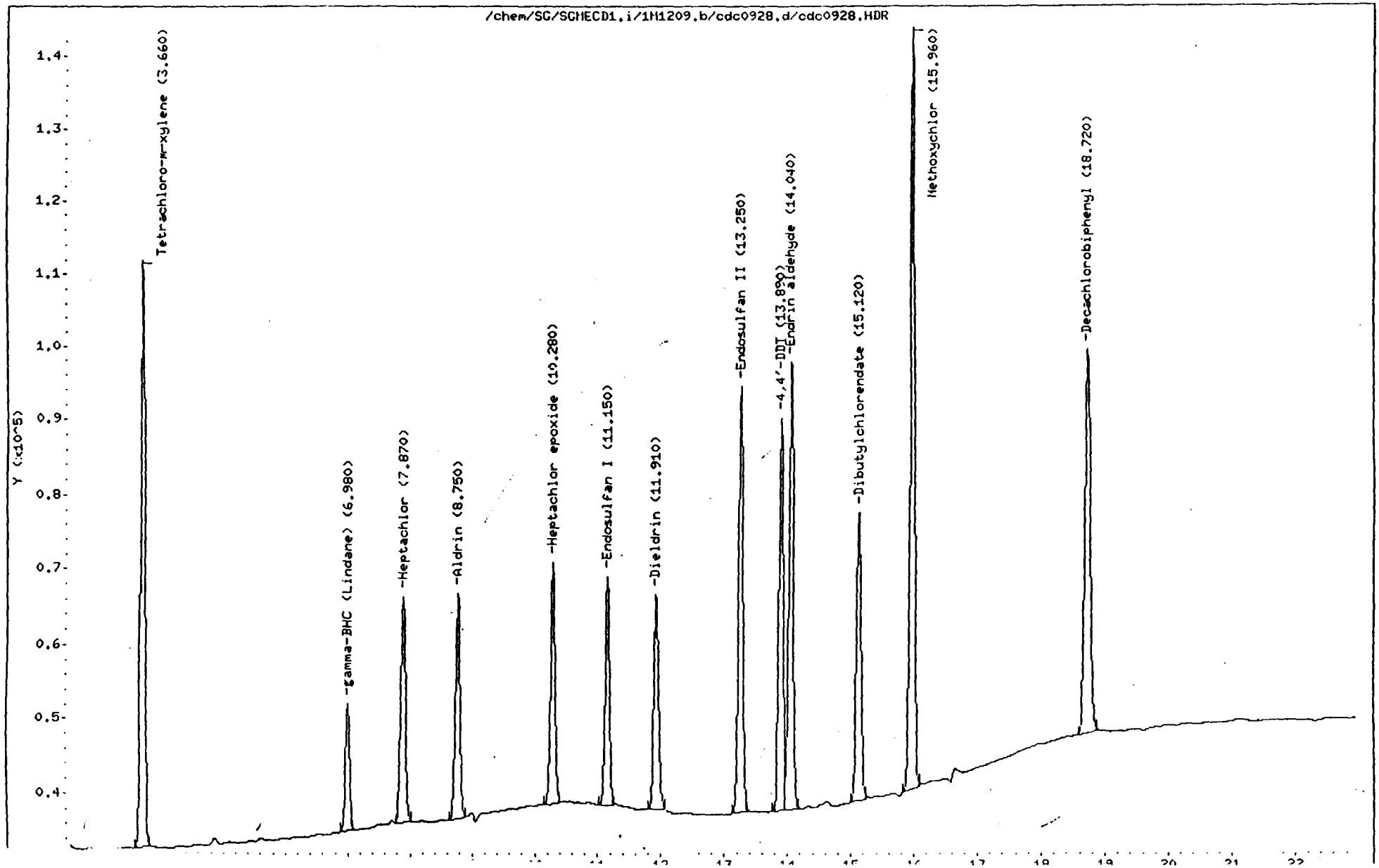
SG45 APPENDIX: EXAMPLE CHROMATOGRAMS

Data File: /chem/SG/SGHECD1.i/1H1209.b/cdc0928.d
Date : 10-DEC-97 07:51:00
Client ID: PISHA-04
Sample Info: PISHA-04~1H1209

Instrument: SGHECD1.i

Operator: BW
Column diameter: 0.53

Column phase: DB-608



Data File: /chem/SG/SGHECD2.i/1H1209.b/ddc0928.d

Date : 10-DEC-97 07:51:00

Client ID: PISHA-04

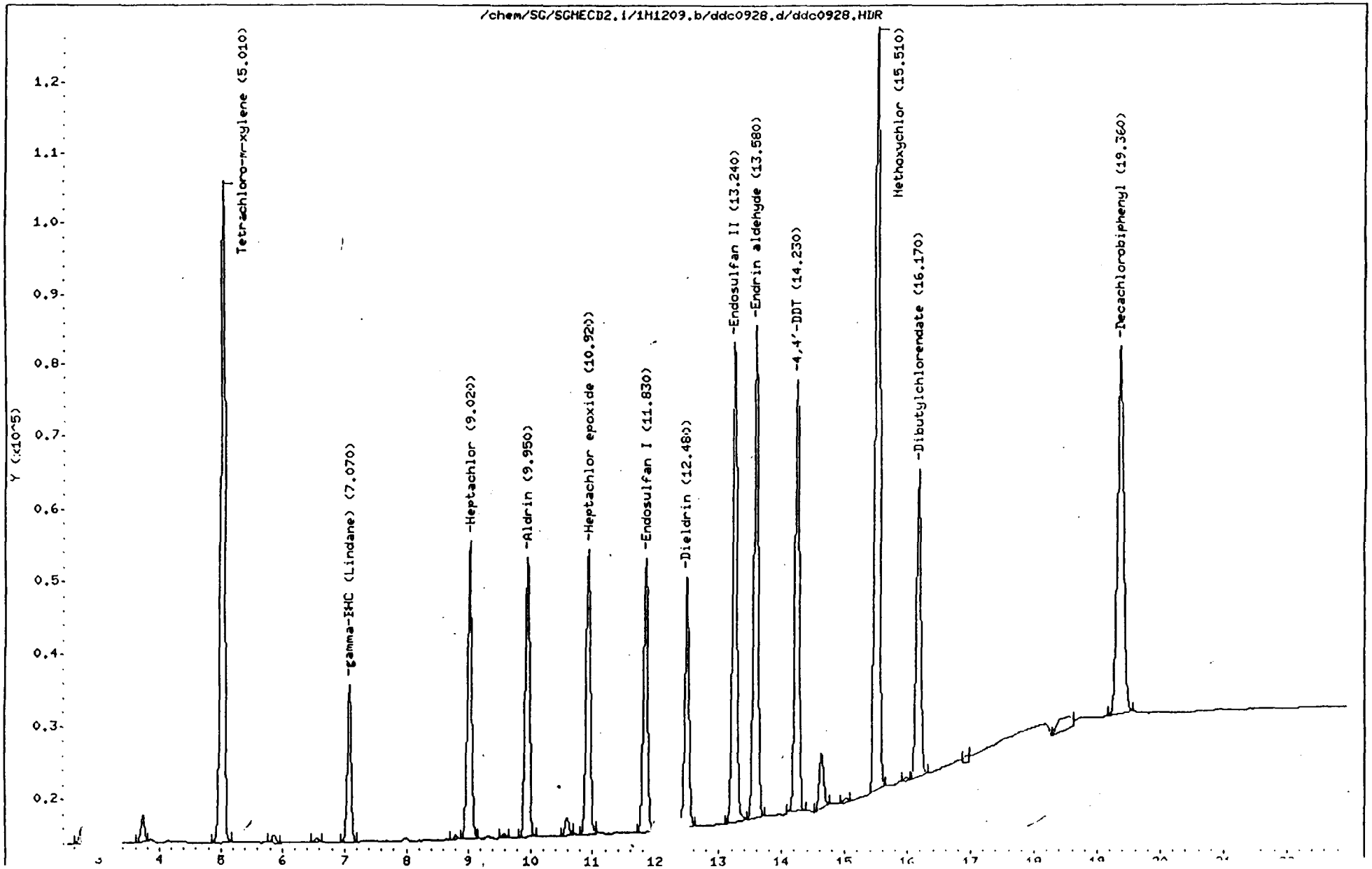
Sample Info: PISHA-04~1H1209

Column phase: DB-5

Instrument: SGHECD2.i

Operator: BW

Column diameter: 0.53

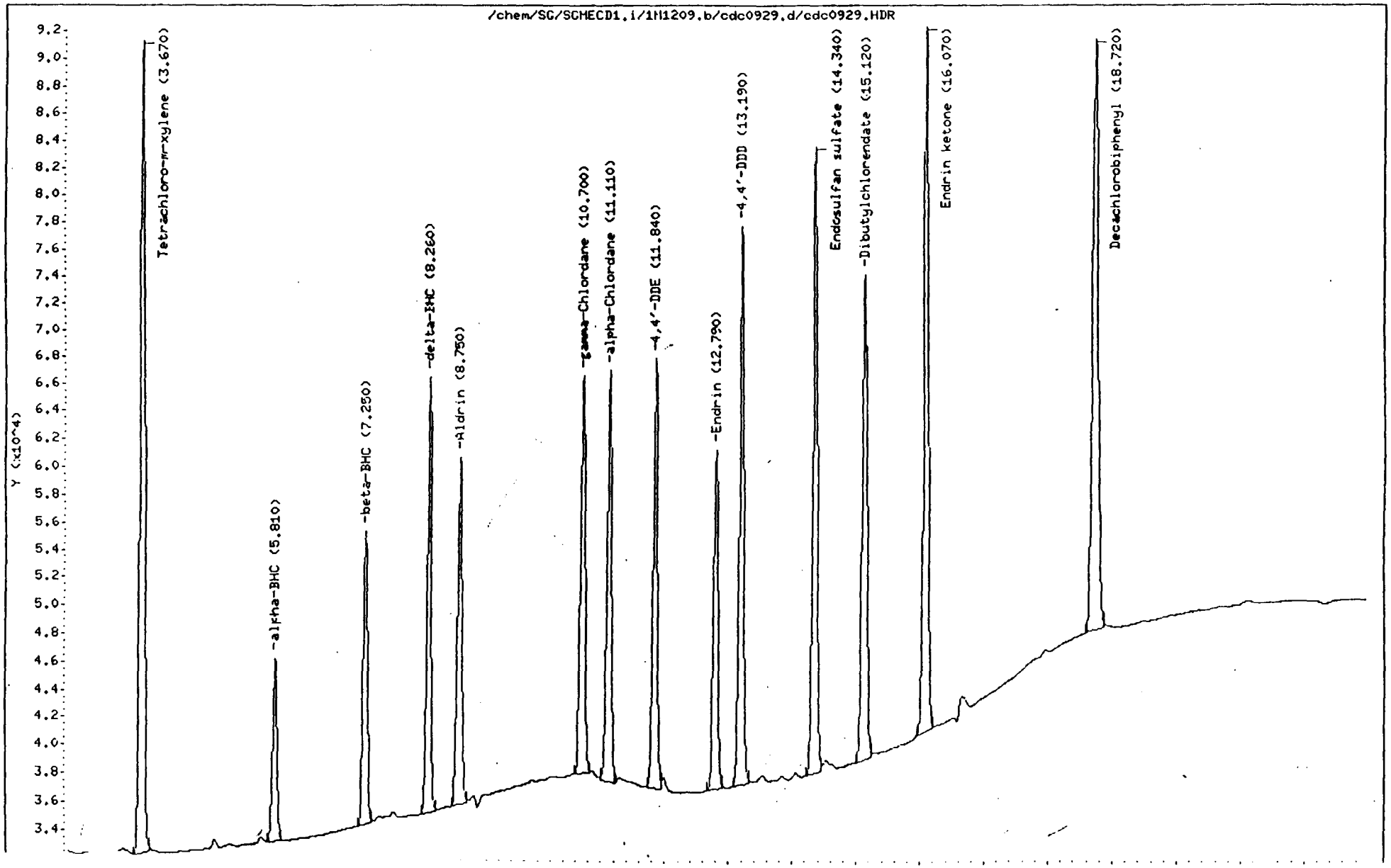


Data File: /chem/SG/SGHECD1.i/1H1209.b/cdc0929.d
Date : 10-DEC-97 08:19:00
Client ID: PISHB-04
Sample Info: PISHB-04~1H1209

Column phase: DB-608

Instrument: SGHECD1.i

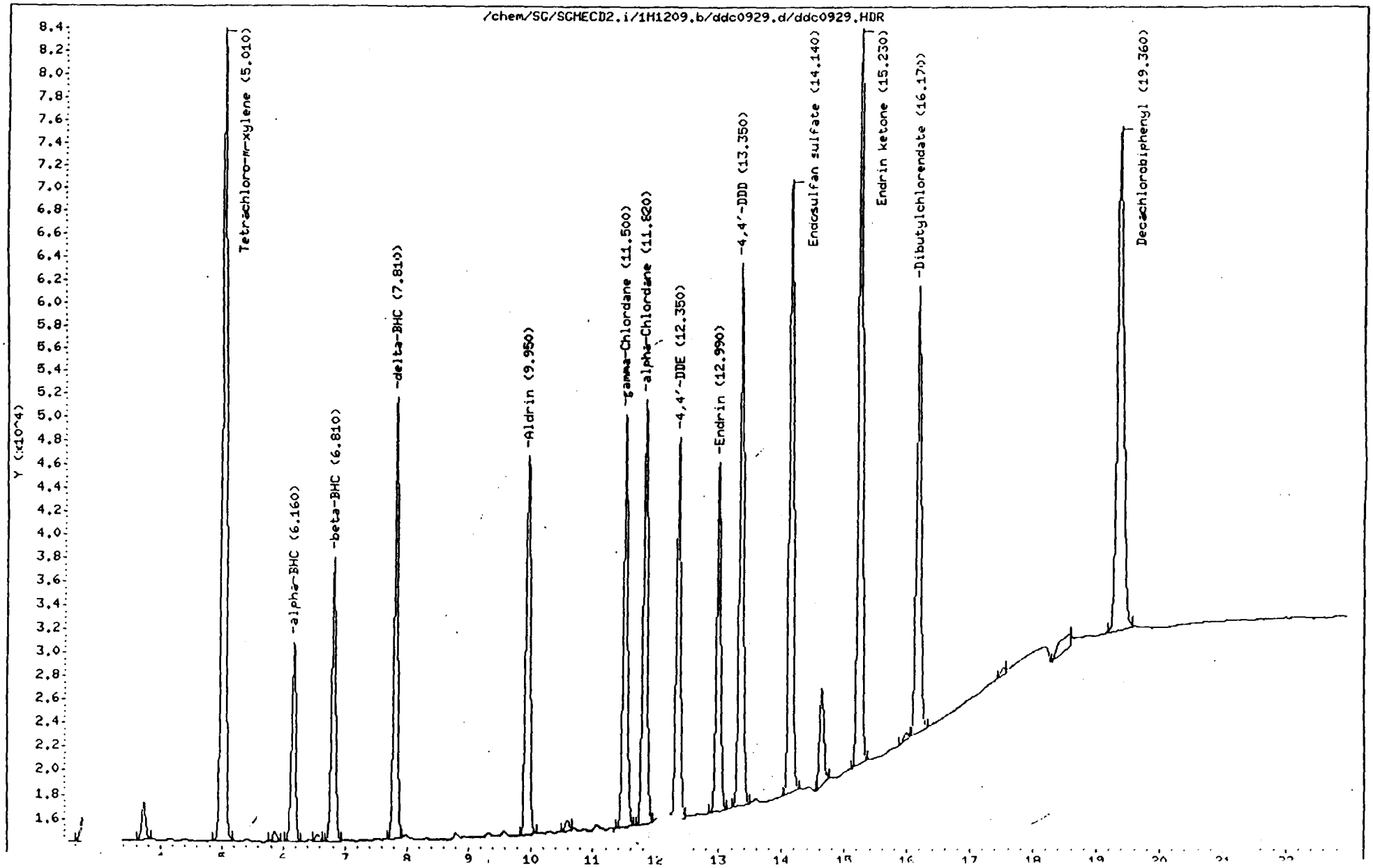
Operator: BW
Column diameter: 0.53



Data File: /chem/SG/SGHECD2.i/1H1209.b/ddc0929.d
Date : 10-DEC-97 08:19:00
Client ID: PISHB-04
Sample Info: PISHB-04~1H1209

Instrument: SGHECD2.i

Operator: BW
Column phase: DB-5
Column diameter: 0.53



Data File: /chem/SG/SGJECD1.i/1J1212.b/edc1244.d

Date : 14-DEC-97 04:22:00

Client ID: PISOC-04

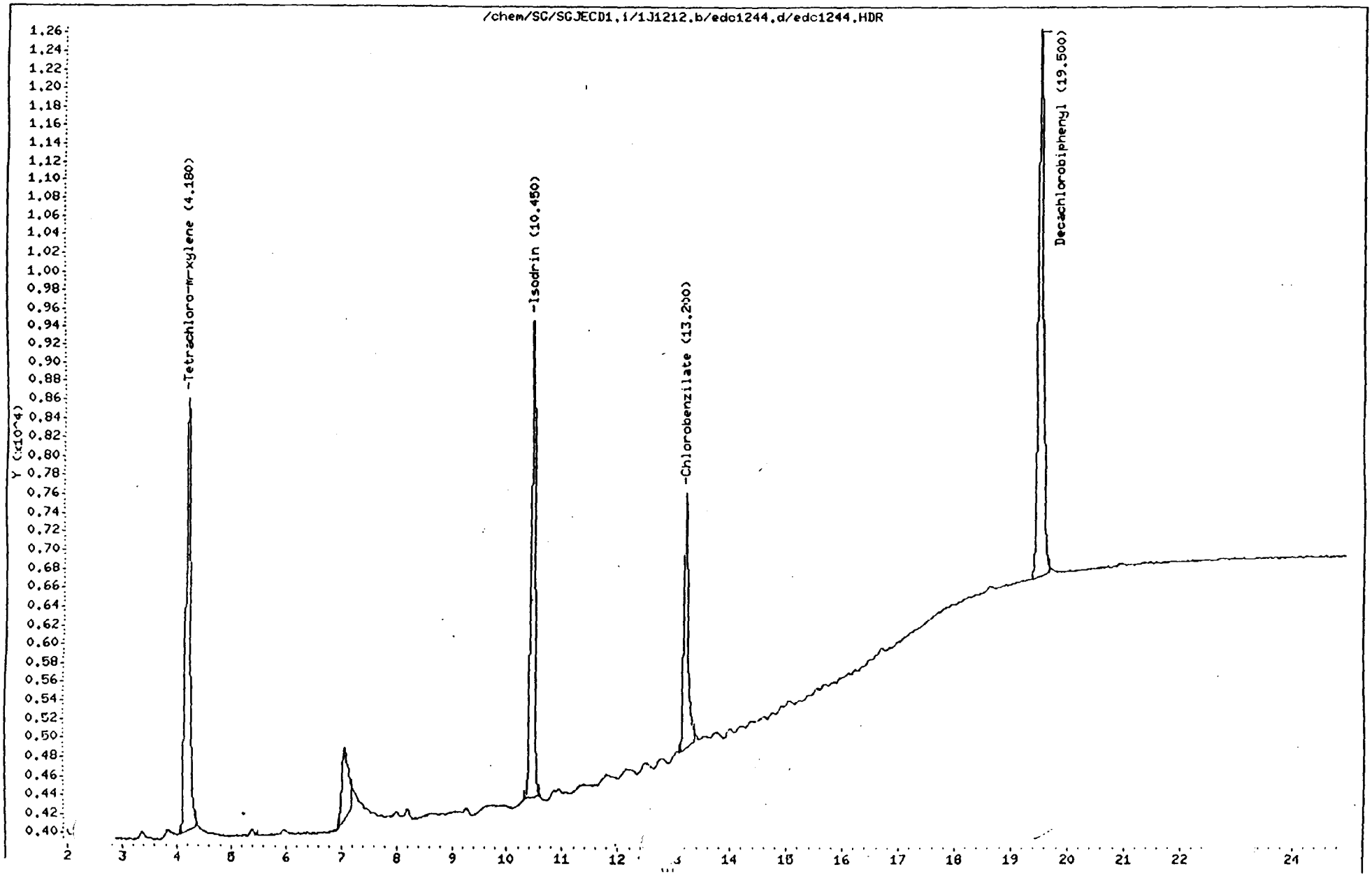
Sample Info: PISOC-04~1J1212

Column phase: DB-608

Instrument: SGJECD1.i

Operator: BW

Column diameter: 0.53



Data File: /chem/SG/SGJECD2.i/1J1212.b/fdc1244.d

Date : 14-DEC-97 04:22:00

Client ID: PISOC-04

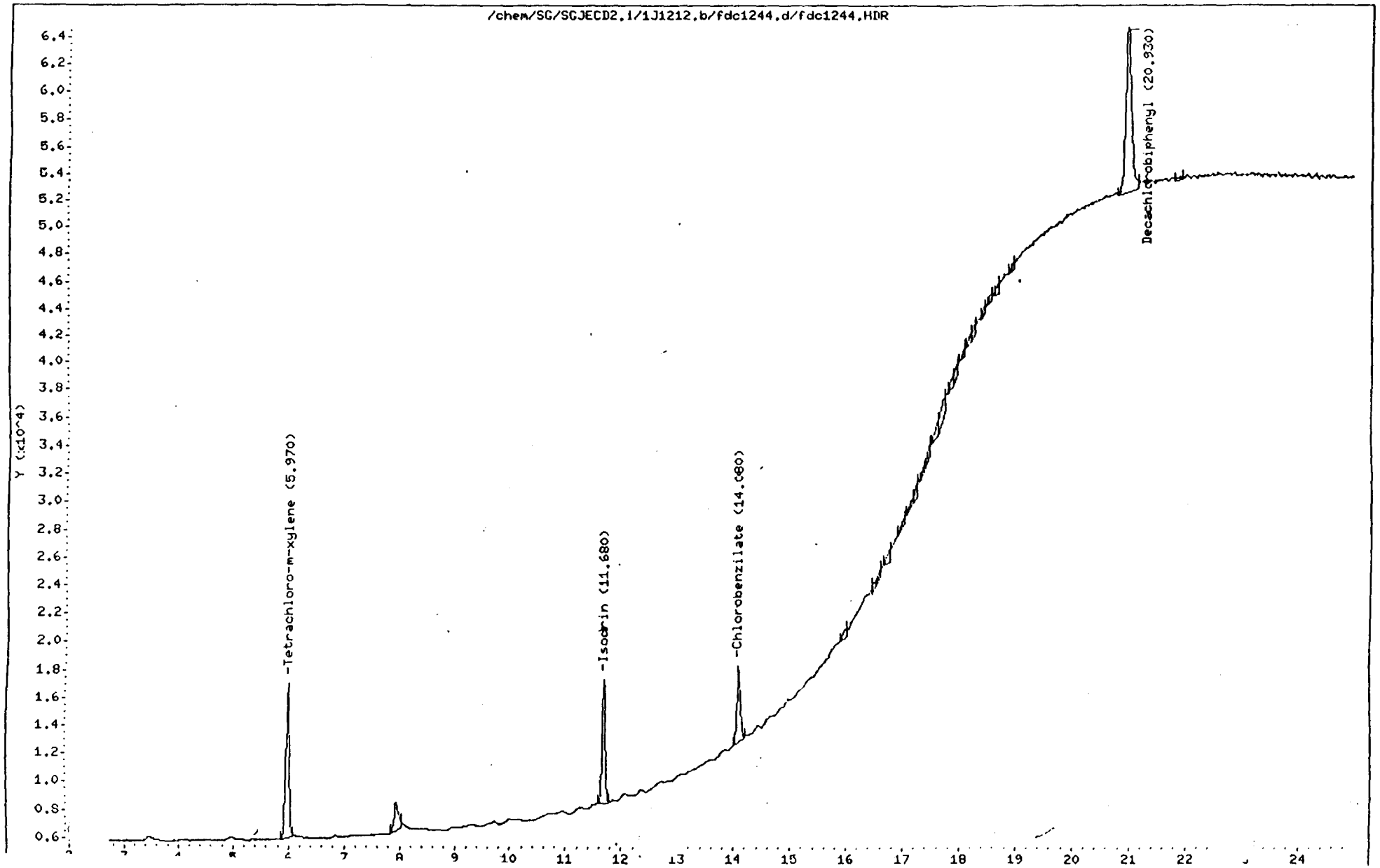
Instrument: SGJECD2.i

Sample Info: PISOC-04~1J1212

Operator: BW

Column phase: DB-5

Column diameter: 0.53



Data File: /chem/SG/SGHECD1.i/1M1209.b/cdc099.d

Date : 09-DEC-97 23:04:00

Client ID: CHLOR-04

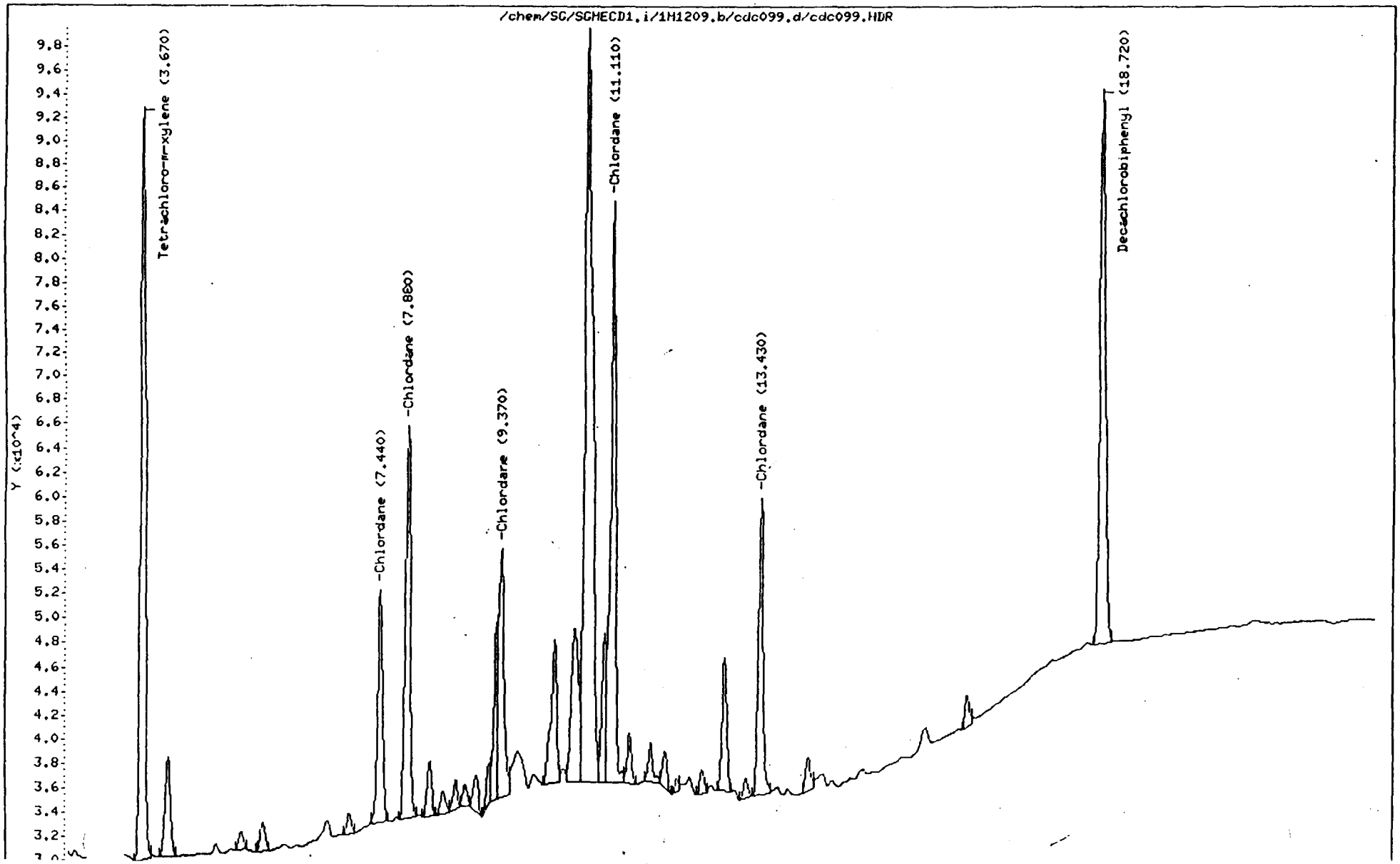
Sample Info: CHLOR-04~1M1209

Column phase: DB-608

Instrument: SGHECD1.i

Operator: BW

Column diameter: 0.53



Data File: /chem/SG/SGMECD2.i/1M1209.b/ddc099.d

Date : 09-DEC-97 23:04:00

Client ID: CHLOR-04

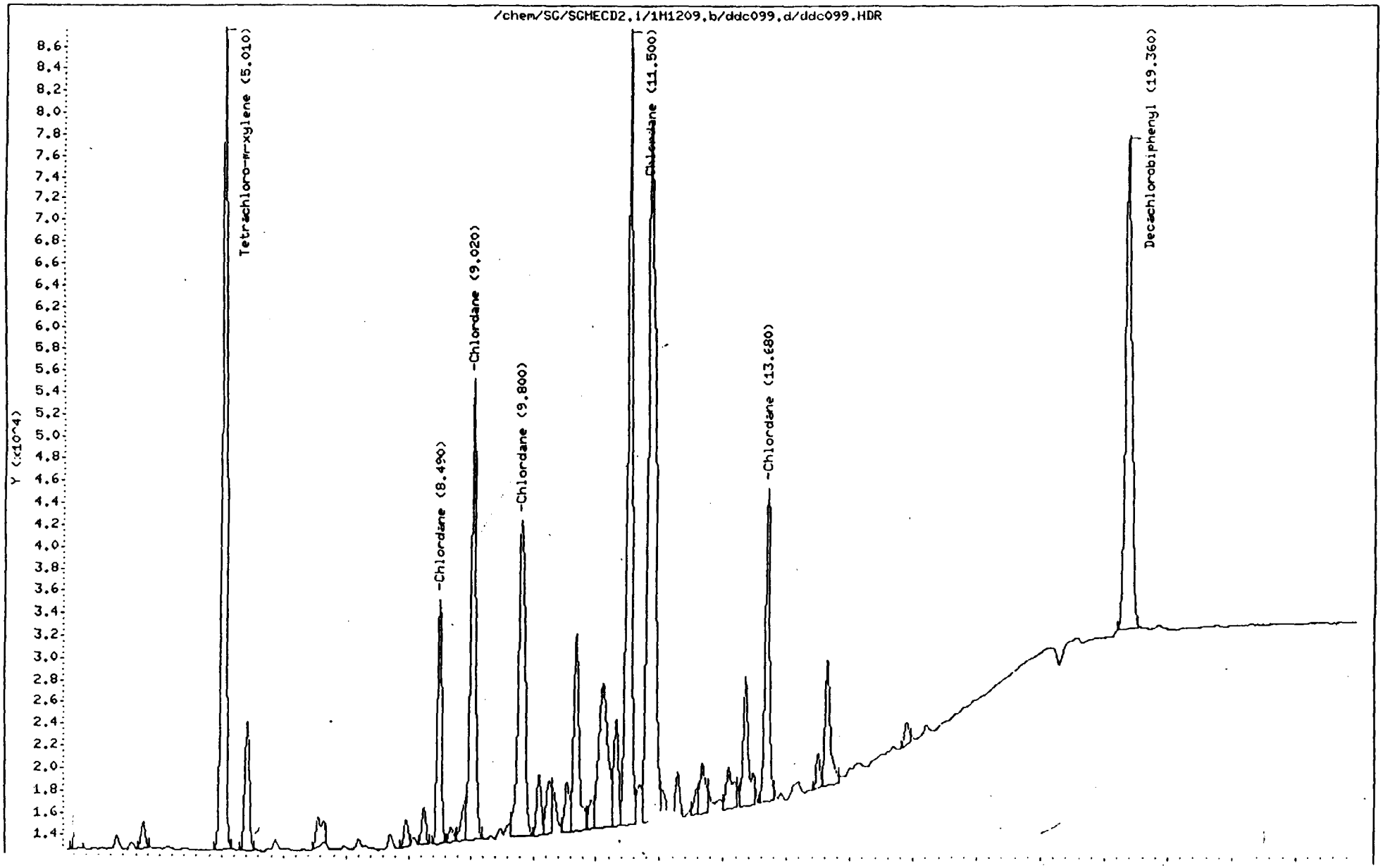
Sample Info: CHLOR-04~1M1209

Column phase: DB-5

Instrument: SGMECD2.i

Operator: BW

Column diameter: 0.53

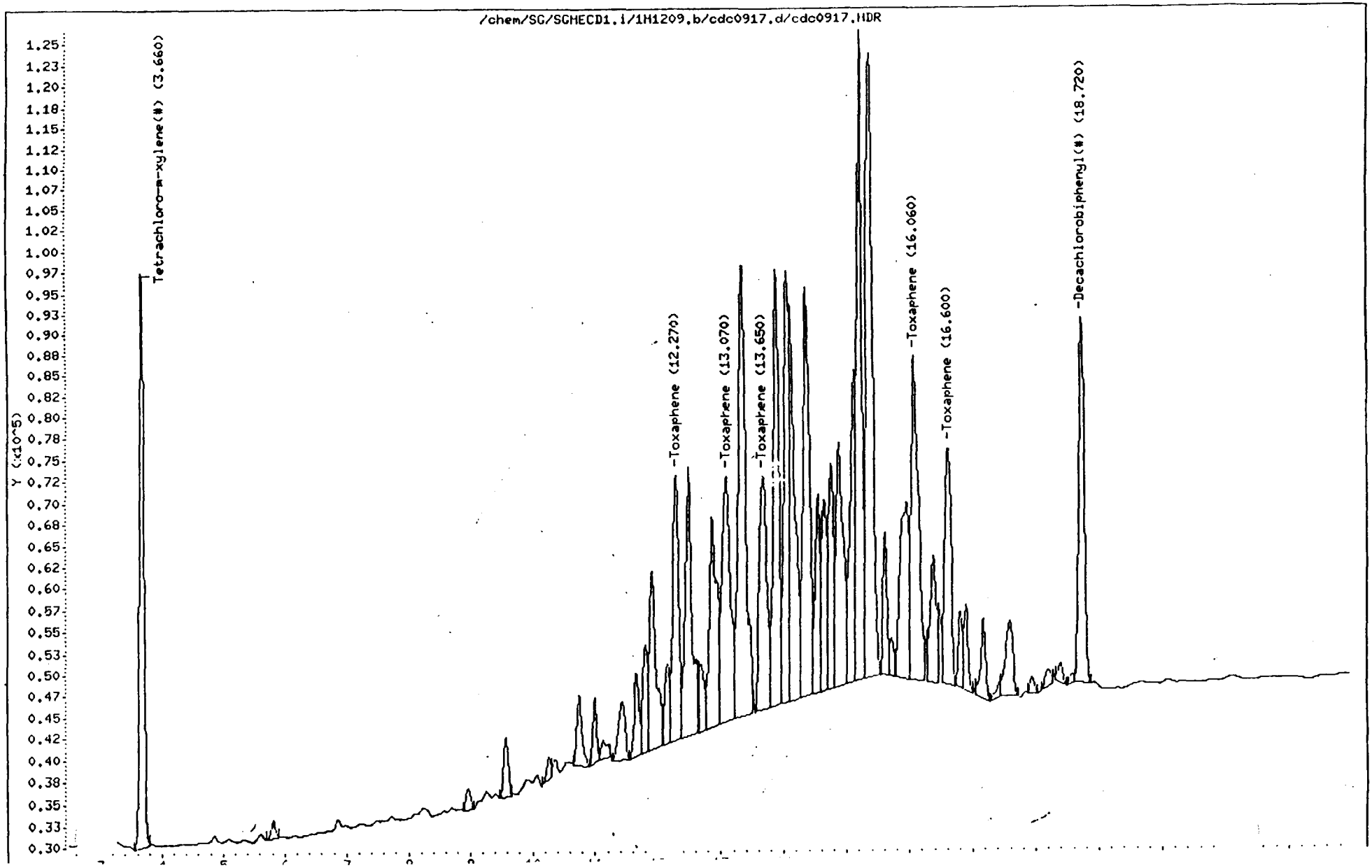


Data File: /chem/SG/SGHECD1.i/1M1209.b/cdc0917.d
Date : 10-DEC-97 02:46:00
Client ID: TOXAPH-04
Sample Info: TOXAPH-04~1M1209

Column phase: DB-608

Instrument: SGHECD1.i

Operator: BW
Column diameter: 0.53



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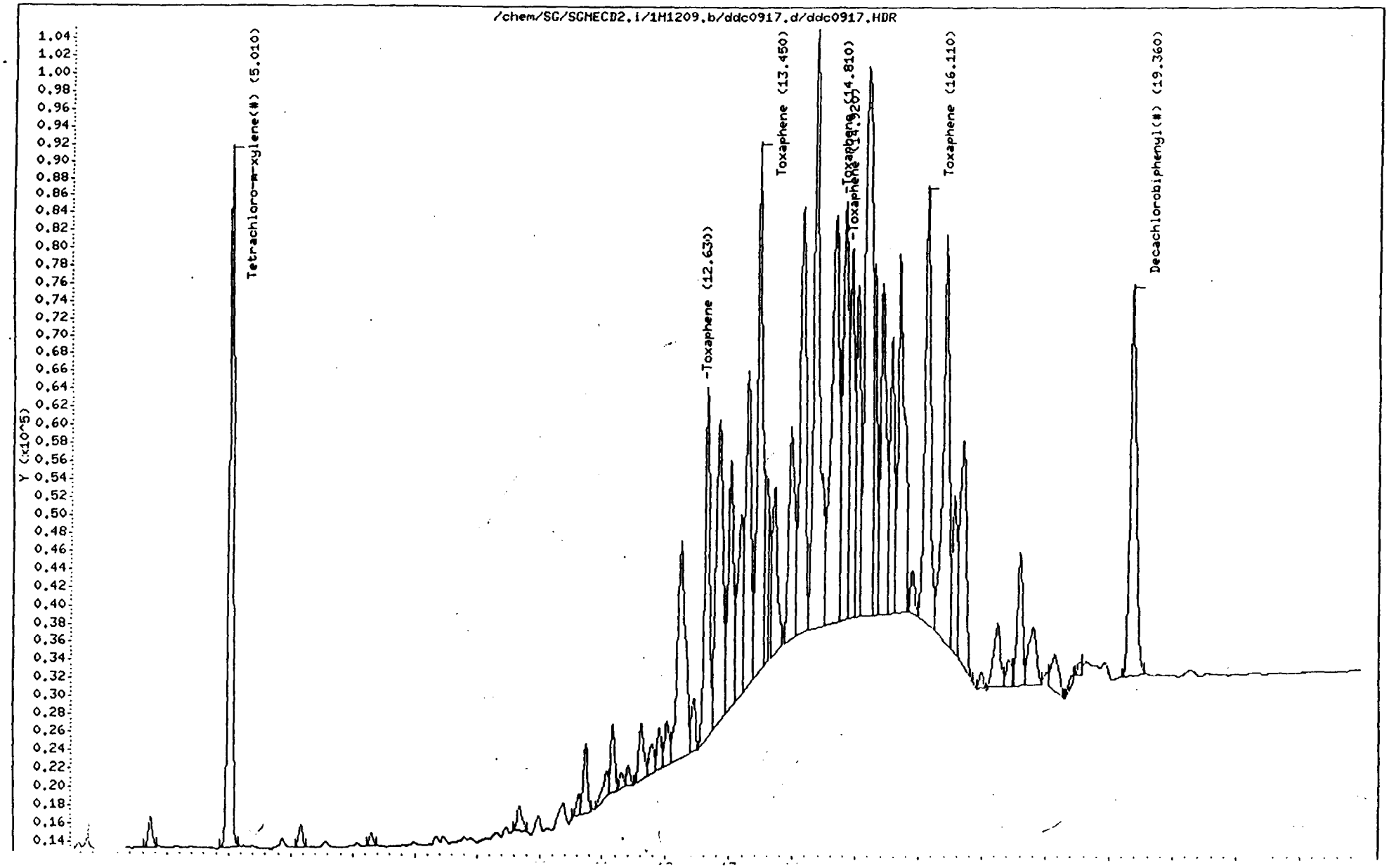
Date : 10-DEC-97 02:46:00
Client ID: TOXAPH-04
Sample Info: TOXAPH-04~1H1209

Instrument: SGHECD2.i

Operator: BW
Column diameter: 0.53

Column phase: DB-5

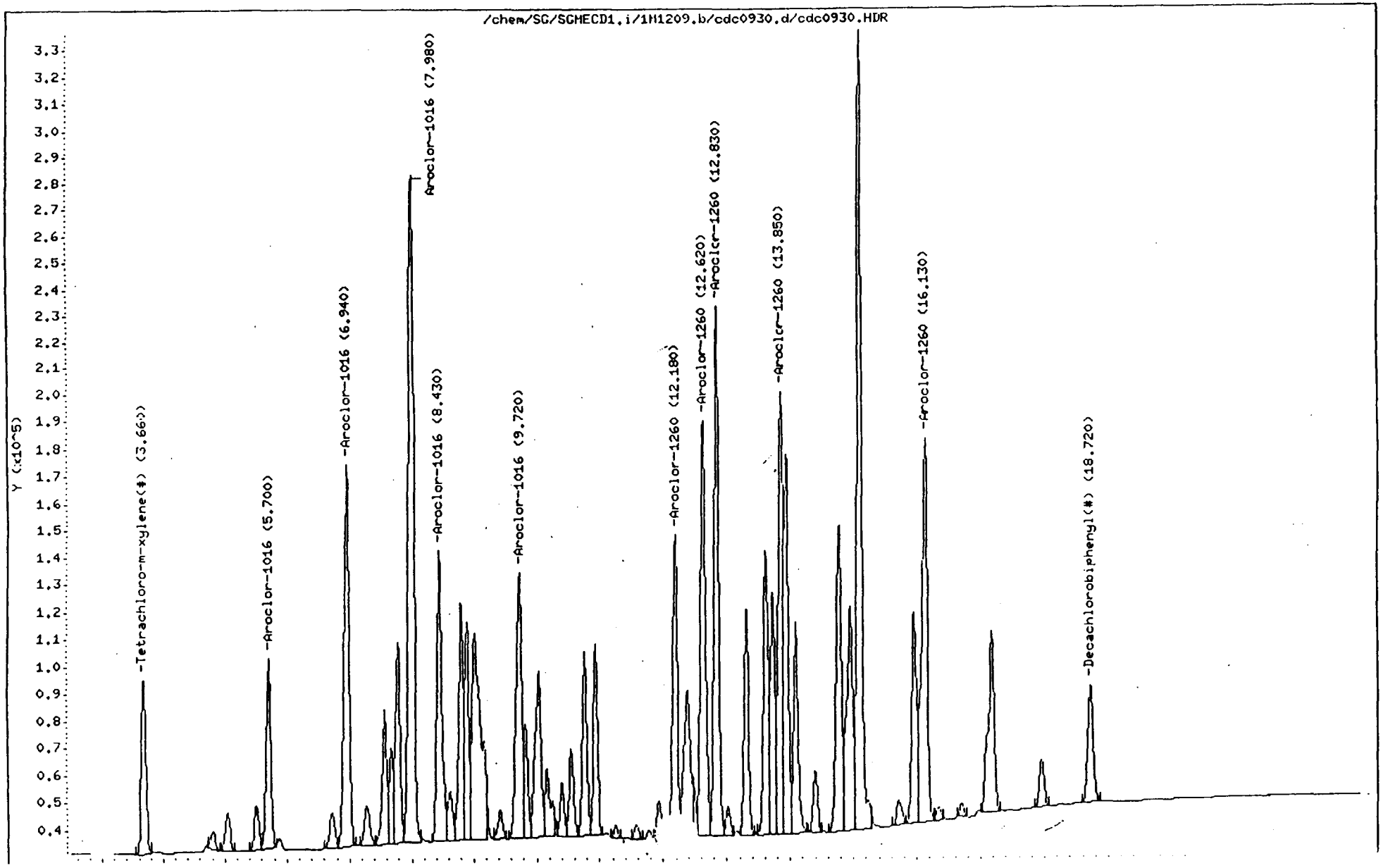


Data File: /chem/SG/SGHECD1.i/1M1209.b/cdc0930.d
Date : 10-DEC-97 08:47:00
Client ID: AR1660-04
Sample Info: AR1660-04^1M1209

Column phase: DB-608

Instrument: SGHECD1.i

Operator: BW
Column diameter: 0.53



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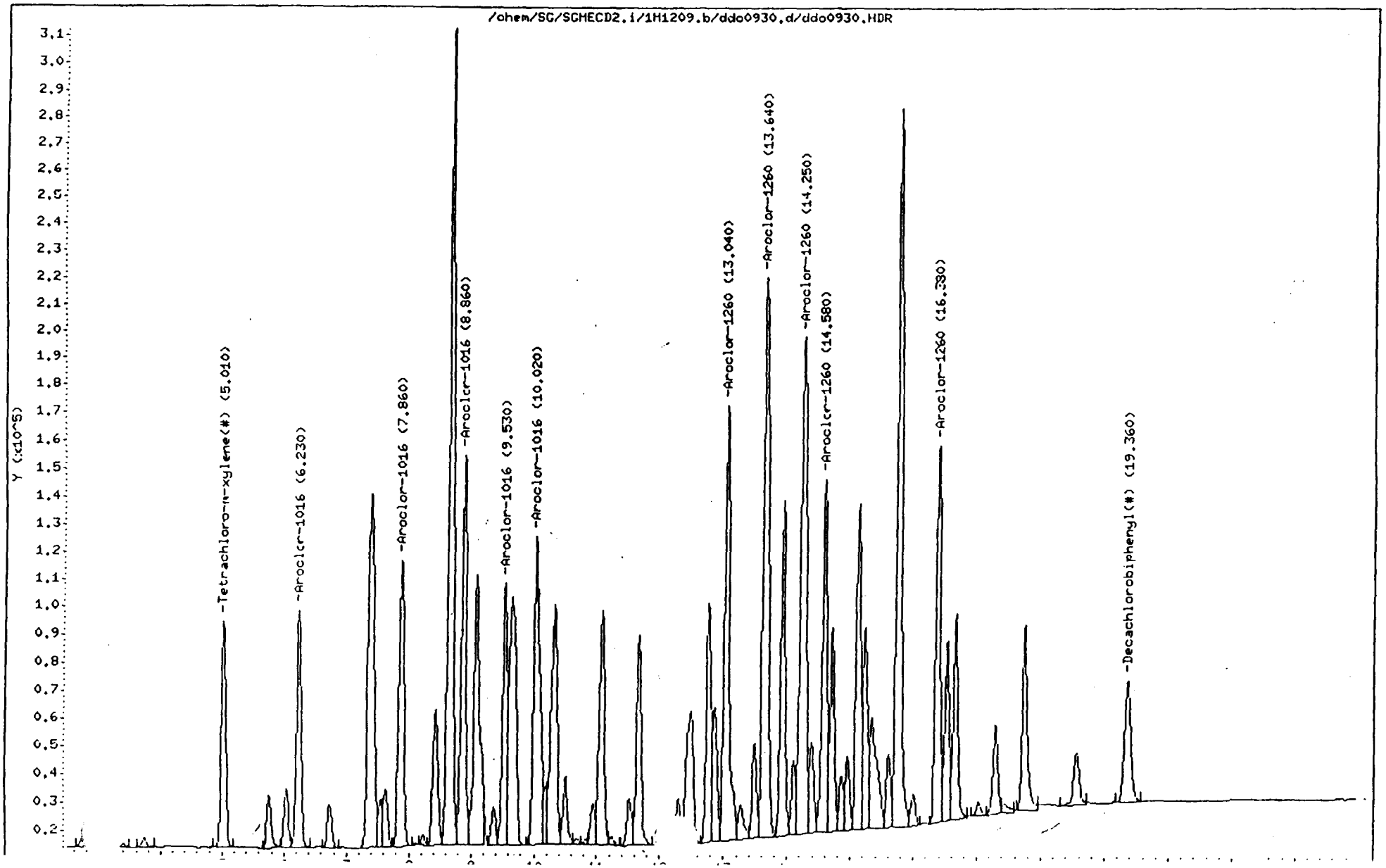
Date : 10-DEC-97 08:47:00
Client ID: AR1660-04
Sample Info: AR1660-04~1H1209

Instrument: SGHECD2.I

Operator: BW
Column phase: DB-5
Column diameter: 0.53



Data File: /chem/SG/SGHECD1.i/1M1209.b/cdc092.d

Date : 09-DEC-97 19:49:00

Client ID: AR1221-04

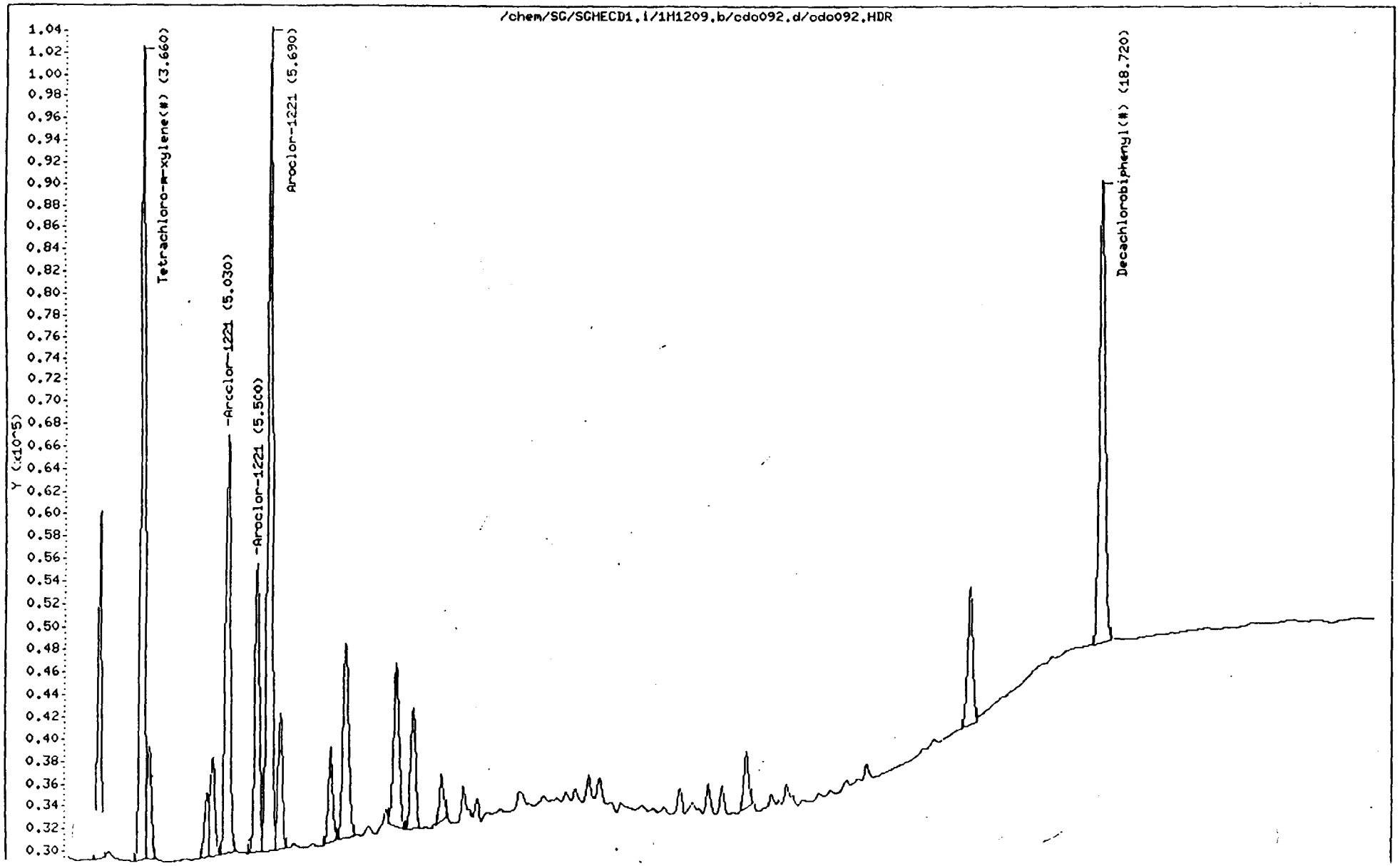
Sample Info: AR1221-04~1M1209

Column phase: DB-608

Instrument: SGHECD1.i

Operator: BW

Column diameter: 0.53



Data File: /chem/SG/SGHECD2.i/1H1209.b/ddc092.d

Date : 09-DEC-97 19:49:00

Client ID: AR1221-04

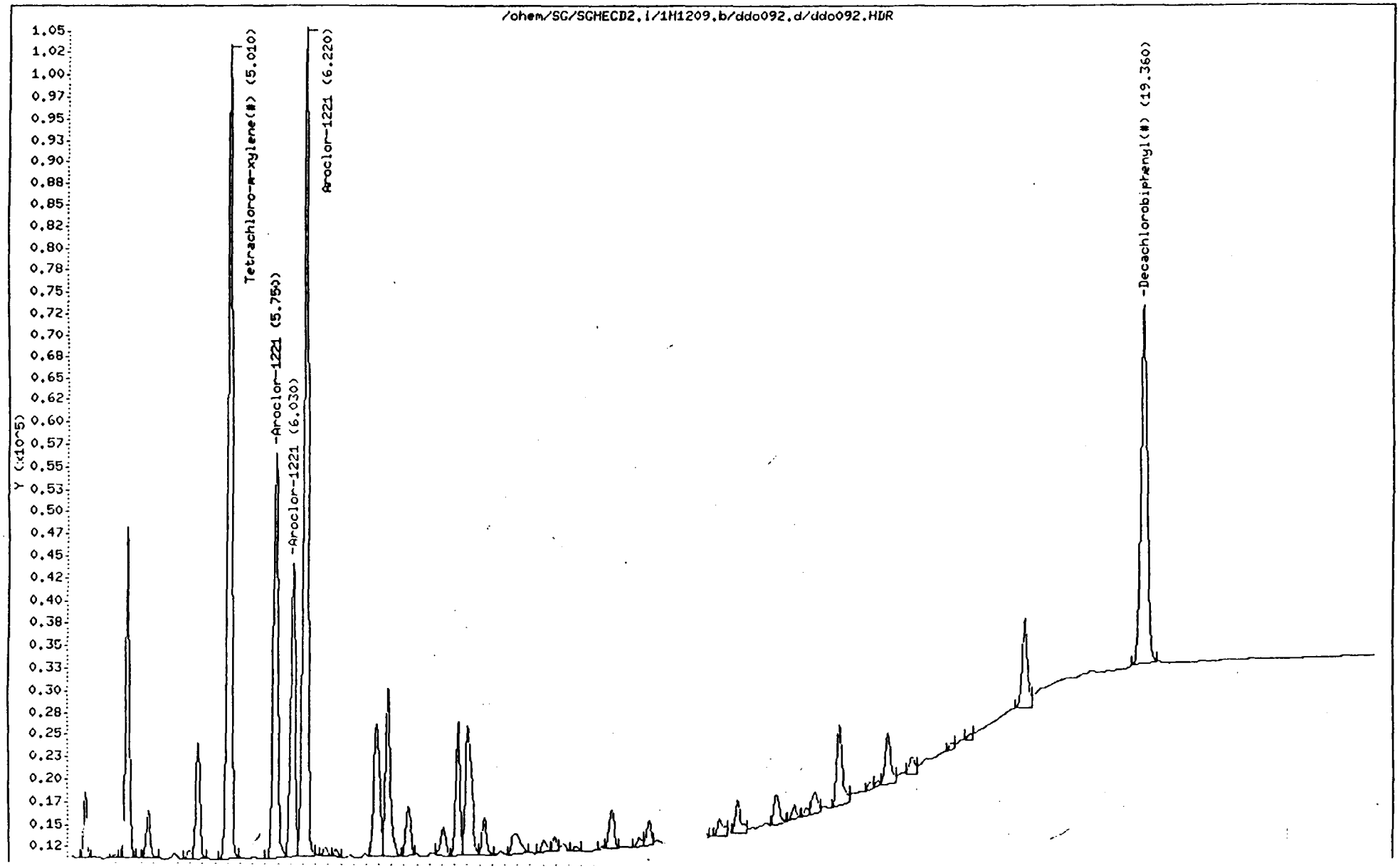
Sample Info: AR1221-04~1H1209

Column phase: DB-5

Instrument: SGHECD2.i

Operator: BW

Column diameter: 0.53

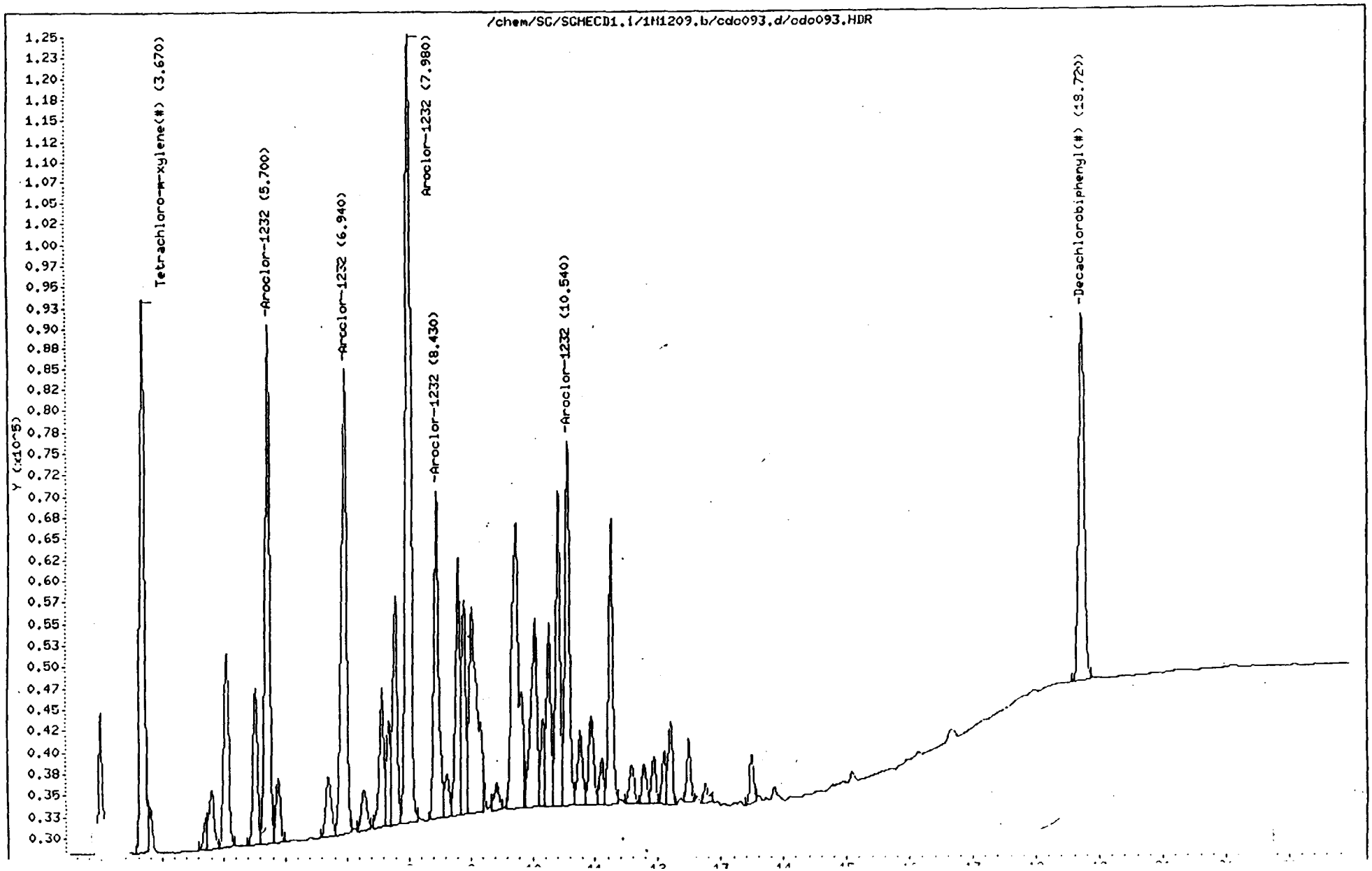


Data File: /chem/SG/SGHECD1.i/1H1209.b/cdc093.d
Date : 09-DEC-97 20:17:00
Client ID: AR1232-04
Sample Info: AR1232-04~1H1209

Column phase: DB-608

Instrument: SGHECD1.i

Operator: BW
Column diameter: 0.53

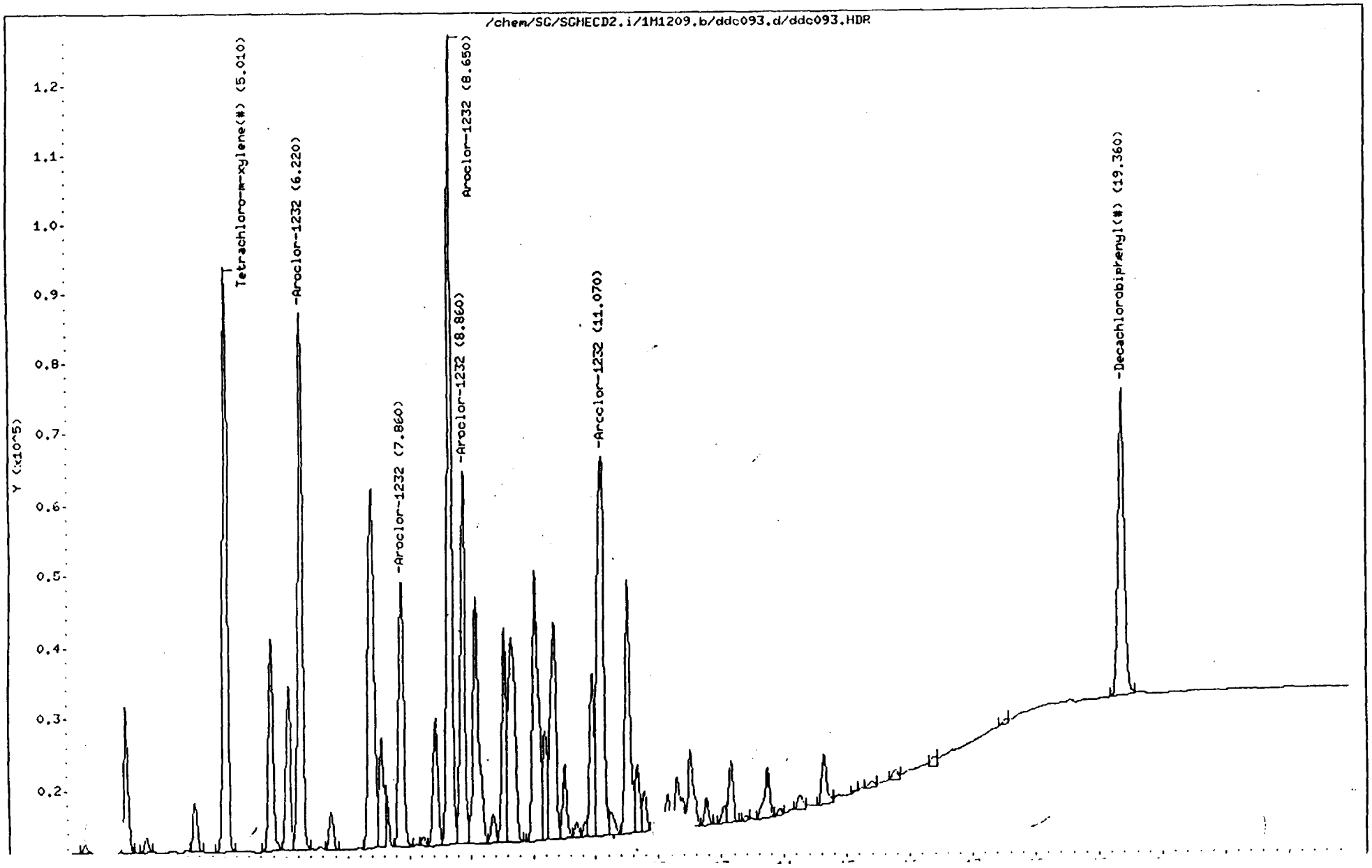


Data File: /chem/SG/SGHECD2.i/1H1209.b/ddc093.d
Date : 09-DEC-97 20:17:00
Client ID: AR1232-04
Sample Info: AR1232-04~1H1209

Column phase: DB-5

Instrument: SGHECD2.i

Operator: BW
Column diameter: 0.53

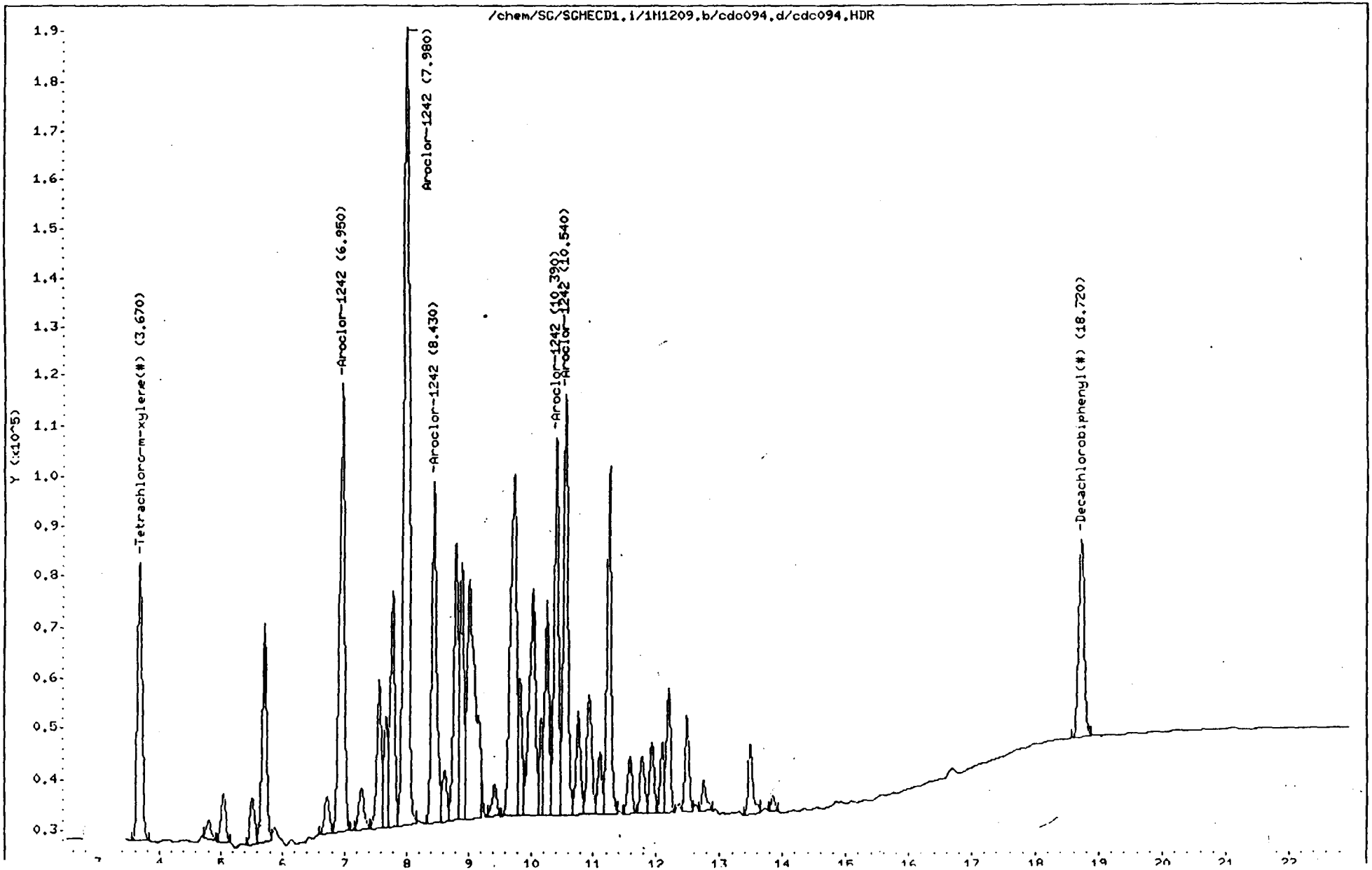


Data File: /chem/SG/SGHECD1.i/1M1209.b/cdc094.d
Date : 09-DEC-97 20:45:00
Client ID: AR1242-04
Sample Info: AR1242-04~1M1209

Column phase: DB-608

Instrument: SGHECD1.i

Operator: BW
Column diameter: 0.53



Data File: /chem/SG/SGHECD2.i/1M1209.b/ddc094.d
Date : 09-DEC-97 20:45:00
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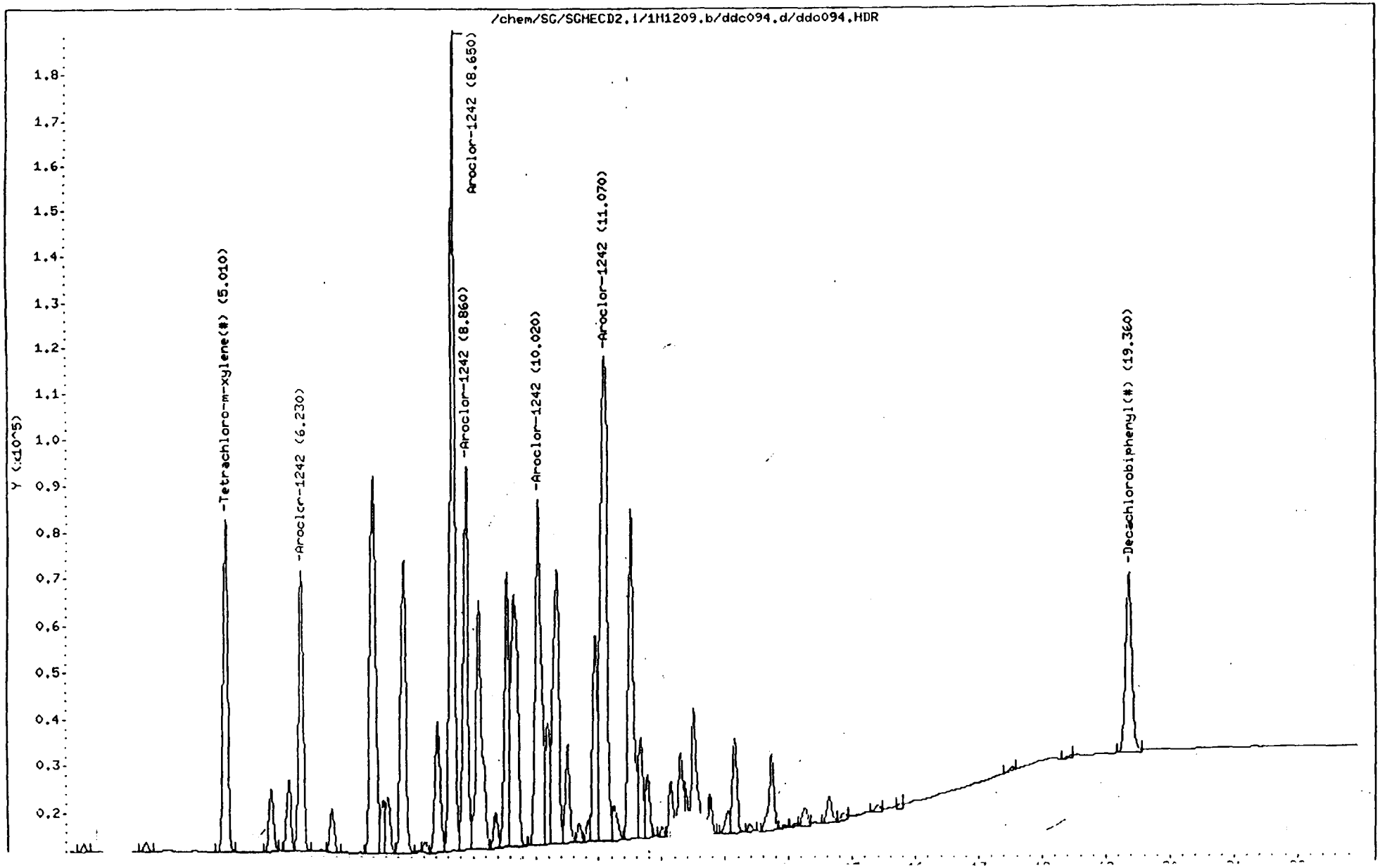
Sample Info: AR1242-04~1M1209

Column phase: DB-5

Instrument: SGHECD2.i

Operator: BW
Column diameter: 0.53



CHANGE -IN-PROGRESS ATTACHMENT

SOP Document No: ME75:12.22.97:3

SOP Description: Graphite Furnace Atomic Adsorption (GFAA)

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager Date: 03/26/98

The following revisions or additions have been made to the referenced SOP.

The SOP Summary (page 14 of 19) is incorrect for LCS for the 200-series methods The recovery limits for LCS are 85-115%. The SOP Summary should read:

QC Item	Frequency	Acceptance Criteria	Corrective Action
Lab Control Sample	One per batch of twenty samples or less	7000-series: SL QAP 200-series: 85-115%	Redigest and reanalyze batch

Approval Signature: R Wayne Robbins R. Wayne Robbins	
Title: Corporate QA Manager	Date: 12/22/97

GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA)

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the determination of metals by graphite furnace atomic absorption (GFAA) spectroscopy. This procedure is applicable to a wide range of matrices including monitoring wells, wastewater, leachates, wastes, soils, and sediments. All matrices, with the exception of some dissolved metals and some drinking water samples, require digestion prior to analysis. Table 1 is a summary of the elements that may be determined using this procedure and some of the parameters for each element.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria are listed in Section 5 of the Savannah Laboratories' Quality Assurance Plans.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be digested using the appropriate preparation method. A representative aliquot of the sample digest is transferred to a graphite tube. The graphite tube is heated in a stepwise manner to dry, char, and atomize the sample. During atomization, the element is volatilized into the optical path where characteristic radiation from a hollow cathode lamp or electrodeless discharge lamp is absorbed. A photosensitive device measures the difference in transmitted radiation as absorbance. Standards and samples are analyzed using the same instrument parameters.

2.2 This SOP is based on the guidance provided in the methods references in Table 2 of this SOP and Method 7000A from SW-846.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do no understand or that will put you or others in potentially dangerous situations.

3.2 The samples have been digested in strong acids and will have high acid concentration. The analyst must wear a lab coat or apron, eye protection (lab glasses or face shield), and gloves when handling samples.

3.3 The GFAA instrument must be properly vented to eliminate metal contamination in the lab.

4.0 INTERFERENCES

4.1 The furnace procedure is subject to chemical interferences. The composition of the sample matrix can have major effect on the analysis. Interferences can be overcome using one or more of the following procedures:

1) Successively dilute and reanalyze the sample to eliminate interferences.
2) Modify the sample matrix either to remove the interference or to stabilize the element.
3) Analyze the sample by the methods of standard addition.

Two quality control checks are performed with each batch of samples as check on interferences: 1), a 1:5 dilution and 2) one sample is subjected to post digestion spike.

4.2 Cross-contamination and contamination of the sample can be major sources of error because of the extreme sensitivities achieved with the furnace. The sample preparation and analytical areas should be kept clean and glassware must be properly cleaned. Special attention should be given to the evaluation of reagent blanks. Pyrolytic graphite can become contaminated and several high temperature "burns" may be required to clean the tube before use.

4.3 Gases generated in the furnace during atomization may have molecular absorption encompassing the analytical wavelength. When this occurs, either use background correction or choose an alternative wavelength. When continuum background correction cannot correct for the interference, chemically remove (chelation, precipitation) the analyte or use an alternate background correction.

4.4 Sample containing large amounts of organic materials must be oxidized by acid digestion before analysis. This will minimize broad-based absorption. Interferences from a "smoke-producing" sample digest can be reduced by extending the charring time and/or charring at a higher temperature. Care must be taken to prevent loss of the analyte.

4.5 Anion interference studies indicate that chloride, and to a lesser extent, phosphate and sulfate anions, may interfere with the GFAA analysis. The use of hydrochloric acid, phosphoric acid, and sulfuric acid should be minimized.

4.6 Carbide formation resulting from the chemical environment of the furnace has been observed. When carbides form, the analyte may be released very slowly from the resulting metal carbide as atomization occurs. The use of a pyrolytically coated graphite tube can reduce carbide formation and increase analyte sensitivity.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

5.1 Aqueous Samples

5.1.1 Liquid sample are collected in 250-mL or 500-mL plastic containers. The sample is preserved with HNO_3 to a pH <2. The sample must be digested and analyzed within 6 months of collection.

5.1.2 Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

5.2 Soil/Sediment Samples

Soil and sediment samples are collected in 500-mL plastic containers. The sample is cooled to 4C at the time of collection and is stored at 4C +/- 2C until time of digestion and analysis. The sample must be digested and analyzed within 6 months of collection.

5.3 TCLP or EP Toxicity Leachate Samples

The leachate is transferred to a plastic container after the extraction procedure. The sample is preserved with HNO_3 to a pH <2.. The leachate sample must be digested and analyzed within 6 months of collection. If the leachate is to be analyzed for mercury, the leachate is stored at 4C+/-2C until the mercury analysis is completed. Hold time for mercury is 28 days

5.4 Waste Samples

Waste samples are collected in 500-mL plastic containers. The sample must be digested and analyzed within 6 months of collection.

6.0 APPARATUS AND MATERIALS

6.1 Atomic absorption spectrophotometer - single or dual channel, single or dual beam instrument, equipped with temperature programmable furnace and autosampler.

6.2 Hollow cathode lamps

6.3 Electrodeless discharge lamps

6.4 Argon gas supply

6.5 Hydrogen/argon: 5% hydrogen in argon for Method 200.9. This gas mixture will increase signal to noise ratio in high chloride samples.

6.6 Pipetting devices - mechanical devices or volumetric pipettes

6.7 Volumetric flasks - class A or equivalent

7.0 REAGENTS

7.1 Reagent water-ASTM Type I or Type II deionized water.

7.2 Nitric acid - reagent grade or better

7.3 1 + 1 Nitric acid solution

7.4 10% Nitric acid solution

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors or, in a very limited number of applications, from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all neat compounds or stock solutions. All preparation steps must be in accordance with SL SOP AN41: *Standard Materials Traceability*.

NOTE: Many standards are to be prepared every six months "or sooner if needed or required," "if needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

8.1 Stock Standards
Stock standards are purchased as certified solutions at a concentration of 1000mg/L. The lab should obtain two sources of each element that are prepared independently.

8.3 Calibration Standards

Calibration standards are prepared from the intermediate standards. The calibration curve must have a minimum of three (3) standards. The lowest standard must be at the reporting limit(RL) and the other standards will define the working range of the calibration curve. See Table 1 for guidance on the calibration levels for each analyte.

$$V\text{int} = \frac{Ccal \otimes Vcal}{C\text{int}}$$

where
Cint = concentration of the intermediate standard, mg/L
Vint = volume of intermediate required to prepare the calibration standard, mL
Ccal = concentration of the calibration standard to prepare, mL
Vcal = volume of calibration standard to prepare, mL

The following tables provide some examples of the preparation of the calibration standards at concentrations listed in Table 1. Note that the solvent is different for EPA Method 200.9 because HCl is also used in the 200.9 digestion procedures. The designations of the standards (A-1, B-2, etc.) are included to differentiate the standard solutions and are not meant to be proscriptive.

Copper, Chromium, Antimony, Silver

CAL STD	INT STD	Vint(mL)	Vcal(mL)*	Ccal, ug/L(1)	Ccal, ug/L (2)
A-1	A	0.050	100	5.0	0.5
A-2		0.10	100	10	1.0
A-3		0.50	100	50	5.0
A-4		0.10	100	100	10

*1% HNO3 (except for Sb prepped by 3005, then 5% HCl, 2% HNO3) Lithium standard levels are 5, 10, 20, 50
(1) copper, chromium, antimony
(2) silver, cadmium

Arsenic, Selenium, Lead, Thallium

CAL STD	INT STD	Vint(mL)	Vcal(mL)*	Ccal, ug/L(1)
B-1	B	0.050	100	5.0
B-2		0.10	100	10
B-3		0.50	100	50
B-4		1.0	100	100

*1% HNO3
(1) selenium, lead, thallium, arsenic

200.9 Calibration Standards

CAL STD	INT STD	Vint(mL)	Vcal(mL)*	Ccal, ug/L(1)
C-1	200.9	0.050	100	5.0
C-2		0.10	100	10
C-3		0.50	100	50
C-4		0.10	100	100

* 2% HNO3 / 1%HCl
(1) selenium, lead, thallium, antimony, arsenic

200.9 Intermediate

ELEMENT	Cstock	Vstock	Vint*	Cint
Arsenic (As)	1000mg/L	1.0mL	100mL	10mg/L
Selenium (Se)	1000mg/L	1.0mL		10mg/L
Lead (Pb)	1000mg/L	1.0mL		10mg/L
Thallium (Tl)	1000mg/L	1.0mL		10mg/L
Antimony (Sb)	1000mg/L	1.0mL		10mg/L

*2% HNO3 / 1%HCl (if prepped). If turbidity < NTU, samples can be run undigested, then matrix would be 1% HNO3.
Note: Concentrations and volumes may need to be adjusted due to the linearity requirements of Method 200.9.

8.4 Calibration Verification Standards (ICV/CCV)

The initial calibration verification (ICV) standard is prepared in the same manner as the intermediate and calibration standards to give a final concentration equal to the concentrations listed in Table 1. The ICV must be prepared from stock or intermediate standards that were prepared independently of the standards used to prepare the calibration standards. This second source may be from a different vendor or a separate lot from the same vendor. The CCV standard is the mid-level calibration standard.

8.5 Calibration Blanks (ICB/CCV)

The calibration blank is reagent water that contains the same acid concentration as the calibration standards, either 1% HNO3 or 2%HNO3 / 1% HCl. The initial calibration blank (ICV) is analyzed after the ICV and the continuing calibration blank (CCB) is analyzed after the CCV.

9.0 SAMPLE PREPARATION

MATRIX	SOP
Aqueous and leachates	ME60
Soils and solids	ME61
Oils and Wastes	ME61/ ME62

10.0 ANALYTICAL PROCEDURE

10.1 Table 1 and its appendices give the general conditions for performing GFAA analysis. These conditions are for guidance. Each lab must optimize and document the conditions used at that lab. Most instrument information can be retrieved from the GFAA data system and retained as evidence of the conditions used during a given analysis.

10.2 Analytical Sequence

Samples and standards are analyzed in a definite sequence given in the SOP Summary. The sequence continues until all samples are analyzed or until a CCV or CCB fails the acceptance criteria. All sample analyses must be bracketed by acceptable calibration and blank verification standards.

10.2.1 Initial Calibration

The initial calibration is performed prior to sample analyses and a calibration curve is constructed

10.2.2 ICV/ICB

The initial calibration standard (ICV) is analyzed after the initial calibration standards but prior to the samples. The ICV is used to verify that the initial calibration standards were properly prepared and that the GFAA is functioning properly.

The initial calibration blank (ICB) is the analyzed after the ICV but prior to sample analyses as a check on the carryover and sensitivity of the GFAA.

10.2.3 CCV/CCB

The continuing calibration verification (CCV) is analyzed after every 10 samples to verify that the calibration remains valid.

The continuing calibration blank (CCB) is analyzed after the CCV as a check on carryover and instrument sensitivity.

10.2.4 Sample analyses [including batch QC (LCS/MS)]

Samples are reported only if the analysis is bracketed by acceptable calibration verification standards and the absorbance of the sample falls within the working range of the GFAA.

10.2.5 Post Digestion Spike (Analytical Spikes)

The post digestion spike is performed to verify that samples of similar matrix types are free from interferences. Two samples from each batch are spiked after the preparation/digestion.

10.2.5.1 Select two samples from each within a batch for the post digestion spike. For batches with 10 or fewer samples, only 1 spike is necessary.

10.2.5.2 Add a known volume of a spiking solution to a known volume of sample digestate. It is suggested that the volume of spiking solution be 1% of the volume or less of the digestate to minimize the effects of volume on the post-digestion spike. The post spiking solution is prepared at a concentration that will yield a spike concentration 2 times the RL when the sample digestate is spiked. Due to the native levels of lead expected in environmental samples, lead is spiked at 4 times the RL. Note that intermediates A & B (prepared earlier in this procedure) may be used for the spike solution.

The following equation can be used to determine the volume of spiking solution required:
where

$$V1 = \frac{C2 \otimes V2}{C1}$$

C_1 = concentration of spiking solution (mg/L)
V_1 = volume of spiking solution (mL)
C_2 = desired concentration of post digestion spike (mg/L)
V_2 = volume of sample used for post-digestion spike (mL)

10.2.5.3 Analyze the spiked aliquot and an un-spiked aliquot

10.2.5.4 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \otimes 100$$

where

Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = theoretical concentration of spike (ug/L)
(See 10.2.5.2)

10.2.5.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 85-115% recovery.

Result of Post Digestion Spikes	Action
Within 85-115% limits	None
>115% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<85% recovery but >50% recovery	Analyze all associated samples by single point method of standard addition and quantify by using MSA. OR qualify all associated samples on report
<50% recovery	Dilute digestate and repeat spike. Analyze all associated samples by single point MSA.

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high.

10.2.5.6 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative absorbance. The first point in the MSA (un-spiked sample) should be greater than or equal to zero absorbance. Use good judgement when evaluating data where the absorbances are negative. The digestate should be diluted and reanalyzed to determine the extract of the matrix interferences.

10.2.5.7 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, Vx, are taken. One aliquot is spiked with a known concentration, Cs. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The absorbance of both aliquots are measured and the sample concentration, Cx, is calculated:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

where

S1 = absorbance of the spiked aliquot
S2 = absorbance of the un-spiked aliquot

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Aqueous Samples

Aqueous samples are routinely reported in mg/L while the GFAA is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

11.1.1 The concentration of the target analyte in liquid samples is calculated as follows:

$$Concentration\,(mg/L) = ug/L(from\ printout) \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where

F = final volume of the sample digestate (L)-usually 50mL (0.050L)
V = volume of sample digested (L)
DF = dilution factor

11.1.2 The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

$$Concentration(mg/L) = RLqap \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where

RLqap = reporting limit from SL QAP (ug/L)
F = final volume of the sample digestate (L)
V= volume of sample digested (L)
DF = dilution factor

The Table 1 Reporting Limits assumes:

F = 50mL
V = 50mL
DF = 1

11.2 Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the GFAA is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

11.2.1 The concentration of the target analyte in soil and solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = ug/L(from\ printout) \otimes \frac{F}{W \otimes solids} \otimes DF \otimes \frac{1mg}{1000ug}$$

where

F = final volume of the sample digestate (L)
W = volume of sample digested (kg)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

For example, if the percent solids is 85%, the decimal equivalent is 0.85; if the %solids is 100%, the decimal equivalent is 1.0.

11.2.2 The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

$$Concentration(mg/kg, dw) = RL_{qap} \otimes \frac{0.0010kg}{W \otimes solids} \otimes \frac{F}{0.100L} x\ DF$$

where

RLqap = reporting limit from Table 5, SL COMPQAP
W = weight of sample digested (kg)
F = final volume of the sample digestate (L)
V = volume of sample digested (L)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

The SL COMPQAP Table 5 Reporting Limits assumes

F = 0.100L (100mL)
DF = 1
W = 0.0010kg (1.0g)
solids = 1.0

11.3 The concentration of the target analyte in the lab control and matrix spike samples is determined as in Section 11.1. The concentration is compared to the theoretical spike concentration and the percent recovery is calculated.

11.3.1 The theoretical concentration of a lab spike or matrix spike is calculated:

$$Ct = \frac{\text{mass of analyte added to sample}}{\text{volume or weight of sample}}$$

For liquids, this equation expands to:

$$Ct = \frac{Cspike \otimes Vspike}{Vsample}$$

For soils, this equation expands to:

$$Ct = \frac{Cspike \otimes Vspike}{Wsample \otimes solids}$$

where
Cspike = concentration of the spiking solution (mg/L)
Vspike = volume of spiking solution added to sample (L)
Vsample = volume of sample spiked (L)
Wsample = weight of sample spiked (kg)
solids = (percent solids)/100

11.3.2 The percent recovery is calculated:

$$\%recovery(\%REC)\frac{Cms - Cs}{C_T} \otimes 100$$

where

Cms = concentration of the spiked sample (MS/MSD or LCS/LCSD)
Cs = concentration of the un-spiked sample (for LCS/LCSD, let Cs = 0)
C_T = theoretical concentration of the spike

11.3.3 The MS/MSD samples are analyzed in duplicate. The relative percent recovery(%RPD), a measure of precision, is calculated for the MS/MSD pair as follows:

$$\%RPD = \left| \frac{(\%REC1 - \%REC2)}{\frac{(\%REC1 + \%REC2)}{2}} \right| \otimes 100$$

where
%REC1 = percent recovery of the MS or LCS
%REC2 = percent recovery of the MSD or LCSD

If duplicate samples are analyzed, the relative percent difference is calculated:

$$\%RPD = \left| \frac{(C1 - C2)}{\frac{(C1 + C2)}{2}} \right| \otimes 100$$

where
C1 = concentration of the sample
C2 = concentration of the duplicate sample

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 SL SOP AN02: *Analytical Batching,* Table 13.1 of the SL QAP, and the SOP Summary provide guidance on evaluating QC and sample data.

12.2 The method detection limit (MDL) is determined annually in accordance with SL SOP CA90. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP.

12.3 Determination of the Instrument Detection Limit (IDL)
The IDL is defined as three times the standard deviation of seven replicate analyses analyzed over three non-consecutive days. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP.

13.0 TROUBLESHOOTING

No items in this revision.

14.0 PREVENTIVE MAINTENANCE

See Section 10 of the current SL QAP.

15.0 REFERENCES

15.1 Savannah Laboratories' ***Comprehensive Quality Assurance Plan***, and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 ***Methods for Chemical Analysis of Water and Waste***; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.

15.3 ***Test Methods for Evaluating Solid Waste, Third Edition***; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986.

Method Summary - GFAA Analysis

HOLD/STORAGE

Container	Minimum 250mL plastic bottle with a plastic or Teflon-lined lid
Preservative	HNO3 to pH<2 in the field. If dissolved metals are required, filter the samples before preservation.
Storage	Liquids preserved to pH <2 may be stored at room temperature until preparation. Solid samples should be stored at 4C (less than 6C with no frozen samples) from collection until preparation.
Hold Time	Samples may be held for up to six months from the time of collection

SAMPLE PREPARATION

Sample prep is detailed in SL SOPs ME60 and ME61..

ANALYTICAL SEQUENCE

200-series and 7000-series methods
Instrument Start-Up: Turn on the AA furnace according to the manufacturer's recommendations. Allow hollow cathode lamps proper warm-up times. Clean the furnace head and replace the graphite tubes as needed. Align the lamp and furnace as recommended by the instrument manufacturer.
Initial Calibration-minimum of three points with the lowest standard at the required reporting limit or CRDL.
Initial Calibration Verification(ICV)-a mid-level standard prepared from a source independent of the source used to prepare the initial calibration standards
Initial Calibration Blank (ICB)
10 samples
Continuing Calibration Verification(CCV)-mid-level calibration standard
Continuing Calibration Blank(CCB)
10 samples
Continuing Calibration Verification(CCV)
Continuing Calibration Blank(CCB)
10 samples
Continuing Calibration Verification(CCV)
Continuing Calibration Blank(CCB)
10 samples
Continuing Calibration Verification(CCV)
Continuing Calibration Blank(CCB)

The sequence continues until all of the samples are analyzed or until the CCV or CCB fail the acceptance criteria. All sample analyses (including dilutions) must be bracketed by acceptable verification standards and blanks.

QC CRITERIA

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration-minimum three(3) standards and a cal blank. Lowest standard at or near the RL	Daily	Correlation >= 0.995	Recalibrate
Initial Calibration Verification Standard (ICV) –Prepared from independent source.	At the beginning of the analysis	SW846/200-series = within ± 10% 200 series/200.9 = within ± 5%	Recalibrate
Continuing Calibration Verification Standard (CCV)-mid-level standard (may be the same source as the initial calibration standards)	At the beginning and end of the analysis and every 10 samples	200 series/200.9 = within ± 10% SW846 = within ± 20% .	Terminate the analysis, fix the problem and reanalyze the previous 10 samples. .
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the RL/CRDL. 200.9 = absolute value of the blank must be less than the RL	SW846/200-series = terminate the analysis, correct the problem and reanalyze the previous 10 samples.
Lab control samples (LCS)	One per batch of 20 samples or less	Recovery within Section 5 limits in SL QAP or method-defined criteria	Re-digest and reanalyze batch
Method (preparation) blank	One per batch of 20 samples or less	SW-846/200-series:| result| <RL in SL QAP or result <5% of the analyte level in the sample. 200.9: |result |< RL in SL QAP or result < 10% of the analyte level in the sample.	Evalute baseline Reanalyze samples for blank Re-digest and reanalyze batch
MS/MSD - SW846	One set per batch of twenty samples or less	Recovery within Section 5 limits in SL QAP	Flag and report data
Detection Limit Check Solution-CLP	At the beginning of the analysis but not before the ICV	%Rec 50-150%	Terminate analysis, fix problem, reanalyze solution.
Post-Digestion Spike	Two samples per batch.	Follow guidance in Section 10.2.5 of this SOP.	Follow guidance in Section 10.2.5 of this SOP.
Method Detection Limit(MDL)	See CA90	Evaluate according to SL SOP CA90	Evaluate according to SL SOP CA90

TABLE 1

Element	Wavelength (nm)	Calibration Curve (ug/L)***	ICV/CCV Conc. (ug/L)	DL Std. Conc. (ug/L) Not incuding 200.9	Post Digestion Spike(ug/L)	LCS + Matrix Spike Conc (mg/L)	
						Water (ug/L)	Soil (mg/kg)
Selenium (Se)	196.0	5, 10, 50, 100	50	5/10	20	50	5.0
Arsenic (As)	193.7	5, 10, 50, 100	50	10	20	50	5.0
Lead (Pb)	283.3	3, 10, 50, 100	50	3(CLP)/5	20	50	5.0
Antimony (Sb)	217.6	5, 10, 50, 100	50	10	20	50	5.0
Thallium (Tl)	276.8	3, 10, 50, 100	50	2/10	20	50	5.0
Cadmium (Cd)	228.8	0.5, 1, 5, 10	5.0	1	2	5	0.50
Silver (Ag)	328.1	0.5, 1, 5, 10	5.0	1	2	5	0.50
Chromium (Cr)	357.9	5, 10, 50, 100	50	10	20	50	5.0
Copper (Cu)	327.4	5, 10, 50, 100	50	10	20	50	5.0
Nickel (Ni)	232.0	5, 10, 50, 100	50	10	20	50	5.0
Lithium (Li)	670.8	5, 10, 20, 50	25	5	10	25	2.5

*If a lower RL is required, the lowest standard must be at or near the RL and the calibration criteria met.
**The DL std is not required if included in the initial calibration for 200-series and 700-series methods. This standard is required for CLP work.
***200.9 standard levels are different for Table 1 due to linearity criteria.

TABLE 2-Recommended Matrix Modifiers

PARAMETER	METHOD	MODIFIER(S)
Antimony (GFAA)	7041(3005)	F(Varian)
	7041(3010)	F(Varian)
	7041(3050)	F(Varian)
	200.9	L, J(Varian)
	204.2	F(Varian)
Arsenic (GFAA)	7060(3020)	H(TJA), I(Varian)
	7060(3050)	H(TJA), I(Varian)
	200.9	L, J(Varian)
	206.2	H(TJA), I(Varian)
	3113B	
Beryllium (GFAA)	7091(3020)	
	7091(3050)	
	210.2	
Cadmium (GFAA)	7131A(3020)	B(Varian)
	7131A(3050)	B(Varian)
	213.2	B(Varian)
	3113B	
Chromium (GFAA)	7191(3020)	none
	7191(3050)	none
	218.2	none
Copper (GFAA)	3113B	
	7211(3020)	none
	7211(3050)	none
	220.2	none
Iron(GFAA)	236.2	
Lead (GFAA)	7421(3020)	A(TJA), B(Varian)
	7421(3050)	A(TJA), B(Varian)
	200.9	L, K
	239.2	A(TJA), B(Varian)
Lithium (GFAA)	7430	G

PARAMETER	METHOD	MODIFIER(S)
Nickel (GFAA)	7521	none
	249.2	none
	3113B	
Selenium (GFAA)	CLP ILMO 2.1/3.0/4.0	Liquid: C(TJA), I (Varian) SS:H(TJA), I(Varian)
	7740(3020)	H(TJA), I(Varian)
	7740(3050)	H(TJA), I(Varian)
	7742(3020)	
	7742(3050)	
	200.9	L, J(Varian)
	270.2	H(TJA), I(Varian)
	3113B	
Silver (GFAA)	7761	B(Varian)
	7761(3050)	B(Varian)
	272.1	B(Varian)
	272.2	B(Varian)
Thallium (GFAA)	CLP ILMO 2.1/3.0/4.0	A(TJA), B(Varian) (J,L used if chloride is at high concentration in sample)
	7841(3020)	A(TJA), B(Varian) (J,L used if chloride is at high concentration in sample)
	7841(3050)	A(TJA), B(Varian) (J,L used if chloride is at high concentration in sample)
	279.2	A(TJA), B(Varian) (J,L used if chloride is at high concentration in sample)
	200.9	J, L
Zinc (GFAA)	3111B	

NOTE: No entry in the modifier column means that there is no current suggestion for a modifier for that parameter.

TJA= Thermo-Jarrel Ash GFAA/Varian = Varian GFAA

TABLE 3 - GFAA MODIFIERS		
Modifier Designation	**Modifier**	**Preparation**
A	10% Ammonium phosphate	50g (NH4)2 HPO4 to 500mL reagent water
B	2% Ammonium phosphate	2g (NH4)2 HPO4 to 100mL reagent water
C	5% Nickel nitrate	24.78g Ni(NO3)2-5H2O to 100mL reagent water
D	1% Nickel nitrate	24.78 Ni(NO3)2-6H2O to 500mL reagent water
E	0.1% Nickel nitrate	2.48g (Ni(NO3)2-6H2O to 500mL reagent water
F	3% Ammonium nitrate	3.0g NH4NO3 to 100mL in 50% HNO3
G	0.3% Ammonium nitrate	0.30g NH4NO3 to 100mL in 50% HNO3
	4% Magnesium reagent	4.21g (Mg(NO3)2-6H2O to 100mL in reagent water
	2% Palladium nitrate	Purchased from CPI
H	1% Pd/2% Mg	50mL 4% magnesium reagent and 50mL 2% palladium nitrate
I	0.04% Pd/0.08% Mg	4mL modifier to 100mL in 1% HNO3
J	0.067% Pd/0.3% Mg	1.3mL 4% magnesium reagent and 0.6mL 2% Palladium nitrate to 20mL with 1% HNO3
K	0.012% Pd/ 0.06% Mg	4mL modifier J to 20mL in reagent water
L	5% Hydrogen in Argon	Purchase from gas company (required for 200.9)

ATTACHMENTS
FURNACE TEMPERATURE PROGRAMS

These furnace programs are provided for guidance and are intended to aid the analyst in establishing instrument parameters. The laboratory must document the actual conditions used.

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 08/05/97
BATCH LI03 MOBILE SAMPLES

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	STOP
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 26 Se 200.9

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	5
LAMP CURRENT (mA)	15
SLIT WIDTH (nm)	1.0
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	196.0
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.20

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	30.0	3.0	ALTERNATE	NO
2	400	10.0	3.0	ALTERNATE	NO
3	1200	15.0	3.0	ALTERNATE	NO
4	1200	10.0	3.0	ALTERNATE	NO
5	500	10.0	3.0	NORMAL	NO
6	500	1.0	0.0	NORMAL	NO
7	2700	1.1	0.0	NORMAL	YES
8	2700	2.0	0.0	NORMAL	YES
9	2700	2.0	3.0	NORMAL	NO
10	40	13.3	3.0	NORMAL	NO

Elements : Ni

M : Single Beam Absorption

Integ Type : Peak Area Integ Time : 6.0 Sec. Delay Time : 0.0 Sec.
Graph Scale: Automatic
Comment : NO MODIFIER/ COATED NON-PLATFORM

Furnace Information:

	Dry	Pyro1	Pyro2	Atom	Clean
Temp	150	500	900	2600	2600
Ramp	2	10	10	0	-
Hold	5	15	5	4	3
Purge	Low	Medium	Low	Off	Medium

Integ Stage : Atom Air Ash : Off Auto-Baseline Corr.? Yes Economizer? No
Deposit Time: 5.0 Delay Time: 10 Auto-Deposit Adjust? No

Element : Ni
Element Name : Ni
Wavelength : 232.00
Bandpass : 0.15
High Voltage : 700
BKG Method : SH
Lamp Current : norm: 5.0 bkg: 3.0
Signif Figs : 5
P t Units : ppb
P c Limits Low : 0
t Limits High: 0

Stdzn Method: Multipoint Stdn.

	Std Names	Std Conc	Abs.
A/Z#1:	BlankSTD	= 0.00	= 0.0000
#2:	5.00STD	= 5.00	= 0.0222
#3:	10.00STD	= 10.00	= 0.0622
A/C#4:	50.00STD	= 50.00	= 0.3020
#5:	100.00STD	= 100.00	= 0.5688

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

SAMPLER PARAMETERS

VOLUMES (ƒL)

	SOLUTION	BLANK	MODIFIER
ANK	--	20	5
STANDARD 1	20	0	5
STANDARD 2	20	0	5
STANDARD 3	20	0	5
STANDARD 4	20	0	5
SAMPLE	20	0	5

RECALIBRATION RATE 0

RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO

TEMPERATURE 90

INJECT RATE 5

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	50
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (ƒL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	89 TO 111
QC SPIKE RATE	4
QC SPIKE VOLUME (ƒL)	8
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	1.6 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

Zeeman Se (NiNO3 mod.) SW 846/200 series

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR	RR
DATE	03/03/97
BATCH	SE

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 8 Se PARR NINO3

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	5
LAMP CURRENT (mA)	6
SLIT WIDTH (nm)	1.0
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	196.0
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.20

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	15.0	3.0	NORMAL	NO
2	300	10.0	3.0	NORMAL	NO
3	300	10.0	3.0	NORMAL	NO
4	900	10.0	3.0	NORMAL	NO
5	900	10.0	3.0	NORMAL	NO
6	900	2.0	0.1	NORMAL	NO
7	2400	1.0	0.0	NORMAL	YES
8	2400	2.5	0.0	NORMAL	YES
9	2600	2.0	3.0	NORMAL	NO
10	40	13.3	3.0	NORMAL	NO

SAMPLER PARAMETERS

VOLUMES (ƒL)

	SOLUTION	BLANK	MODIFIER
ANK	--	15	3
STANDARD 1	15	0	3
STANDARD 2	15	0	3
STANDARD 3	15	0	3
STANDARD 4	15	0	3
SAMPLE	15	0	3

RECALIBRATION RATE 0

RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO

TEMPERATURE 100

INJECT RATE 5

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20	
QC INITIAL STANDARD POSITION	45	
QC INITIAL BLANK POSITION	51	
QC CONTINUING STANDARD POSITION	50	
QC CONTINUING BLANK POSITION	51	
QC STANDARD VOLUME (ƒL)	15	
QC STANDARD CONCENTRATION	1.0	ug/l
QC STANDARD LIMITS (%)	90 TO 110	
QC SPIKE RATE	1	
QC SPIKE VOLUME (ƒL)	6	
QC SPIKE CONCENTRATION	50.0	ug/l
MATRIX SPIKE CONCENTRATION	50.0	ug/l
INSTRUMENT DETECTION LIMIT	1.0	ug/l
REQUIRED DETECTION LIMIT	10.0	ug/l

Zeeman Se(CAPE) 7740

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR MM
DATE 02/25/97
BATCH SE10 0128G MDL'S

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 21 Se CAPE NINO3

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	5
LAMP CURRENT (mA)	10
SLIT WIDTH (nm)	1.0
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	196.0
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.20

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	15.0	3.0	NORMAL	NO
2	300	10.0	3.0	NORMAL	NO
3	400	10.0	3.0	NORMAL	NO
4	1000	10.0	3.0	NORMAL	NO
5	1000	10.0	3.0	NORMAL	NO
6	1000	1.0	0.0	NORMAL	NO
7	2400	1.0	0.0	NORMAL	YES
8	2400	3.0	0.0	NORMAL	YES
9	2600	5.0	3.0	NORMAL	NO
10	40	13.3	3.0	NORMAL	NO

02-25
m

SAMPLER PARAMETERS
VOLUMES (fL)

	SOLUTION	BLANK	MODIFIER
.ANK	--	20	0
TANDARD 1	20	0	0
STANDARD 2	20	0	0
STANDARD 3	20	0	0
STANDARD 4	20	0	0
SAMPLE	20	0	0

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 135
INJECT RATE 7

QC PROTOCOL PARAMETERS

Working Standard Lot # IT-CAPE

Date Prepared 02.25.97

Analyst mm

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	50
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (fL)	20
QC STANDARD CONCENTRATION	50.0 UG/L
QC STANDARD LIMITS (%)	80 TO 120
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	8
QC SPIKE CONCENTRATION	50.0 UG/L
MATRIX SPIKE CONCENTRATION	50.0 UG/L
INSTRUMENT DETECTION LIMIT	1.0 UG/L
REQUIRED DETECTION LIMIT	5.0 UG/L

SAMPLE	CONC UG/L	%RSD	MEAN ABS	READINGS	
BLANK	0.0		0.124	0.122	0.125
QC				25Feb97	12:22
				CONCENTRATION LESS THAN IDL	
STANDARD 1	5.0	0.0	0.018	0.018	0.018
QC				25Feb97	12:26
STANDARD 2	10.0	21.7	0.044	0.051	0.038
QC				25Feb97	12:30
				%RSD GREATER THAN LIMIT	
STANDARD 2	10.0	0.3	0.046	0.046	0.046
QC				25Feb97	12:34
TANDARD 3	50.0	3.6	0.222	0.216	0.228
				25Feb97	12:38
STANDARD 4	100.0	0.8	0.436	0.438	0.434
QC				25Feb97	12:42

Zeeman Se SW846/200 Series

SE06
02.19
LR

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 02/19/97
BATCH SE06 1213E,0123L,0213G

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 3 Se CLP PDMG

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	5
LAMP CURRENT (mA)	15
SLIT WIDTH (nm)	1.0
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	196.0
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.20

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	25.0	3.0	NORMAL	NO
2	300	10.0	3.0	NORMAL	NO
3	400	10.0	3.0	NORMAL	NO
4	1100	10.0	3.0	NORMAL	NO
5	1300	10.0	3.0	NORMAL	NO
6	1300	1.0	0.0	NORMAL	NO
7	2500	1.0	0.0	NORMAL	YES
8	2500	2.0	0.0	NORMAL	YES
9	2600	2.0	3.0	NORMAL	NO
10	40	13.3	3.0	NORMAL	NO

SAMPLER PARAMETERS

VOLUMES (µL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	20	5
STANDARD 1	20	0	5
STANDARD 2	20	0	5
STANDARD 3	20	0	5
STANDARD 4	20	0	5
SAMPLE	20	0	5

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 90
INJECT RATE 5

QC PROTOCOL PARAMETERS

Working Standard Lot # FT

Date Prepared 02.19.97

Analyst LR

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (µL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (µL)	12
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	1.0 ug/L
REQUIRED DETECTION LIMIT	5.0 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.0		-0.000	0.000	-0.001
QC				19Feb97	07:33
				CONCENTRATION LESS THAN IDL	
STANDARD 1	5.0	3.1	0.019	0.019	0.018
QC				19Feb97	07:38
STANDARD 2	10.0	2.1	0.047	0.048	0.046
QC				19Feb97	07:42
STANDARD 3	50.0	0.4	0.198	0.198	0.199
QC				19Feb97	07:47
STANDARD 4	100.0	2.3	0.371	0.365	0.377
QC				19Feb97	07:52

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 08/05/97
BATCH LI03 MOBILE SAMPLES

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	STOP
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 13 As 200.9

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	7
LAMP CURRENT (mA)	9
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	193.7
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	0.95

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	30.0	3.0	ALTERNATE	NO
2	400	10.0	3.0	ALTERNATE	NO
3	1200	15.0	3.0	ALTERNATE	NO
4	1200	10.0	3.0	ALTERNATE	NO
5	500	10.0	3.0	ALTERNATE	NO
6	500	1.0	3.0	ALTERNATE	NO
7	2700	1.1	0.2	ALTERNATE	YES
8	2700	2.0	0.0	ALTERNATE	YES
9	2700	2.0	3.0	NORMAL	NO
10	40	13.3	3.0	NORMAL	NO

SAMPLER PARAMETERS

VOLUMES (ƒL)

	SOLUTION	BLANK	MODIFIER
ANK	--	15	5
STANDARD 1	15	0	5
STANDARD 2	15	0	5
STANDARD 3	15	0	5
STANDARD 4	15	0	5
SAMPLE	15	0	5

RECALIBRATION RATE 0

RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO

TEMPERATURE 90

INJECT RATE 5

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	50
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (ƒL)	15
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	79 TO 121
QC SPIKE RATE	4
QC SPIKE VOLUME (ƒL)	6
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	1.0 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

Zeeman - As (CAPE) liquid 7060

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR MM
DATE 02/25/97
BATCH AS06 0128G MDL'S

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 20 As CAPE NIN03

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	7
LAMP CURRENT (mA)	7
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	193.7
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	0.95

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	10.0	3.0	NORMAL	NO
2	300	15.0	3.0	NORMAL	NO
3	500	15.0	3.0	NORMAL	NO
4	1400	10.0	3.0	NORMAL	NO
5	1400	15.0	3.0	NORMAL	NO
6	1400	3.0	3.0	NORMAL	NO
7	2600	1.0	0.0	NORMAL	YES
8	2600	3.0	0.0	NORMAL	YES
9	2600	5.0	3.0	NORMAL	NO
10	40	13.3	3.0	NORMAL	NO

SAMPLER PARAMETERS
VOLUMES (µL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	20	0
STANDARD 1	20	0	0
STANDARD 2	20	0	0
STANDARD 3	20	0	0
STANDARD 4	20	0	0
SAMPLE	20	0	0

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 135
INJECT RATE 7

QC PROTOCOL PARAMETERS

Working Standard Lot # JT-CAPE

Date Prepared 02.25.97

Analyst mm

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	50
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (µL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	80 TO 120
QC SPIKE RATE	0
QC SPIKE VOLUME (µL)	8
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	1.1 ug/L
REQUIRED DETECTION LIMIT	4.6 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.0		0.004	0.003	0.006
QC				25Feb97	10:05
				CONCENTRATION LESS THAN IDL	
STANDARD 1	5.0	1.4	0.025	0.025	0.025
QC				25Feb97	10:10
STANDARD 2	10.0	4.2	0.056	0.054	0.057
QC				25Feb97	10:14
STANDARD 3	50.0	0.6	0.268	0.269	0.267
QC				25Feb97	10:19
STANDARD 4	100.0	1.5	0.505	0.500	0.510
QC				25Feb97	10:23

Zeeman As (NiNO3 mod.) SW846/200 series

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR RR
DATE 03/03/97
BATCH AS

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 10 As PARR NINO3

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	7
LAMP CURRENT (mA)	7
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	193.7
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	0.95

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	135	10.0	3.0	NORMAL	NO
2	220	10.0	3.0	NORMAL	NO
3	500	10.0	3.0	NORMAL	NO
4	800	10.0	3.0	NORMAL	NO
5	1100	10.0	3.0	NORMAL	NO
6	1100	7.0	3.0	NORMAL	NO
7	1100	1.0	0.0	NORMAL	NO
8	2400	1.0	0.0	NORMAL	YES
9	2400	3.0	0.0	NORMAL	YES
10	2500	2.0	3.0	NORMAL	NO
11	40	12.3	3.0	NORMAL	NO

SAMPLER PARAMETERS

VOLUMES (fL)

	SOLUTION	BLANK	MODIFIER
.ANK	--	15	4
STANDARD 1	15	0	4
STANDARD 2	15	0	4
STANDARD 3	15	0	4
STANDARD 4	15	0	4
SAMPLE	15	0	4

RECALIBRATION RATE 0

RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO

TEMPERATURE 120

INJECT RATE 7

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (fL)	15
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	7
QC SPIKE VOLUME (fL)	6
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	1.5 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

Zeeman As SW 846/200 series

HS05
02-24
R

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR	RR
DATE	02/24/97
BATCH	AS05 0224J

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 2 As CLP PDMG

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	7
LAMP CURRENT (mA)	7
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	193.7
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	0.95

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	135	10.0	3.0	NORMAL	NO
2	220	5.0	3.0	NORMAL	NO
3	500	10.0	3.0	NORMAL	NO
4	1100	15.0	3.0	NORMAL	NO
5	1300	7.0	3.0	NORMAL	NO
6	1300	1.0	0.0	NORMAL	NO
7	2600	1.0	0.0	NORMAL	YES
8	2600	3.0	0.0	NORMAL	YES
9	2700	2.0	3.0	NORMAL	NO
10	40	13.3	3.0	NORMAL	NO

02-24-
RR

SAMPLER PARAMETERS
VOLUMES (fL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	20	5
STANDARD 1	20	0	5
STANDARD 2	20	0	5
STANDARD 3	20	0	5
STANDARD 4	20	0	5
SAMPLE	20	0	5

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 100
INJECT RATE 7

QC PROTOCOL PARAMETERS

Working Standard Lot # IT
Date Prepared 02-24-97
Analyst LR

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (fL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	8
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	1.1 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.0		-0.001	0.001	-0.002
QC				24Feb97	16:24
				CONCENTRATION LESS THAN IDL	
STANDARD 1	5.0	1.3	0.026	0.026	0.026
QC				24Feb97	16:28
STANDARD 2	10.0	0.6	0.055	0.055	0.055
QC				24Feb97	16:33
STANDARD 3	50.0	1.6	0.239	0.236	0.242
QC				24Feb97	16:37
STANDARD 4	100.0	2.1	0.432	0.426	0.439
QC				24Feb97	16:42

Atomizer: Furnace
Matrix: SOIL AND WASTE WATER

TJA 4000 As (CAPE soils) SW846(7060)

..HOD INFORMATION **

Default Setup:

Number of Repeats : 2
Flush Time (sec) : 5.0
Auto-Increment Sample Names? No

Auto-print Calibration Curves? : Yes
Analysis Graphics Display : Aborbances & Temp Profile
Auto-print Analysis Graphics? : No
Auto-save Analysis Graphics? : No

Default File Names:

Analysis Data File : OCT95
Autosampler Table : AUTOTAB
Sample Limits Table : LCTAB
Blank (AZ) Limits Table : BLANKLCT
QC Check Table : QCTAB
Recovery Check Table : RQCTAB

OUTPUT INFORMATION **

..ut Mode: Concentration
..ride Print Limits? Yes
Override Significant Figures? No
..imits Table: LCTAB Check? No
Correction Factor: 1

Auto-print data? Yes
Condensed report format? Yes
Auto-store data? Yes
Store individual repeats? Yes

Report to:
Screen Avgs, Stats, Reps, Checks, Units
Printer Avgs, Stats, Reps, Checks, Units

Elements : As Se

Mode : Single Beam Absorption

g Type : Peak Area Integ Time : 1.5 Sec. Delay Time : 1.2 Sec.
raph Scale: Automatic
Comment : 0.1 mL 5.0% NiNO3 to 5mL Sample - PLATFORM TUBE

Furnace Information:

	Dry	Pyro1	Pyro2	Atom	Clean
Temp	200	900	900	2600	2600
Ramp	3	15	0	0	-
Hold	0	10	2	4	2
Purge	Low	Medium	Low	Off	High

Integ Stage : Atom Air Ash : Off Auto-Baseline Corr.? Yes Economizer? No
Deposit Time: 12.0 Delay Time: 15 Auto-Deposit Adjust? No

Element : As
Element Name : As
Wavelength : 197.20
Bandpass : 1.0
Lamp Pos. : 3
High Voltage : Manual Value: 625
BKG Method : SH
Lamp Current : norm: 3.3 bkg: 2.5
Signif Figs : 5
Print Units : ppb
t Limits Low : 0
t Limits High: 0

Stdzn Method: Multipoint Stdn.

	Std Names	Std Conc	Abs.
A/Z#1:	BlankSTD	= 0.00	= 0.0000
#2:	5.00STD	= 5.00	= 0.0123
#3:	10.00STD	= 10.00	= 0.0252
A/C#4:	50.00STD	= 50.00	= 0.1336
#5:	100.00STD	= 100.00	= 0.2465

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

Element : Se
Element Name : Se
Wavelength : 196.03
Bandpass : 1.0
Lamp Pos. : 2
High Voltage : Manual Value: 650
BKG Method : SH
Lamp Current : norm: 3.1 bkg: 2.0
Signif Figs : 5
Print Units : ppb
Print Limits Low : 0
Print Limits High: 0

Stdzn Method: Multipoint Stdn.

	Std Names	Std Conc	Abs.
	BlankSTD	= 0.00	= 0.0000
	5.00STD	= 5.00	= 0.0148
#3:	10.00STD	= 10.00	= 0.0257
/C#4:	50.00STD	= 50.00	= 0.1165

Stdzn Method: Standard Additions

Addn Name	Std Addn
#1: ADD1	= 10.00
#2: ADD2	= 20.00
#3: ADD3	= 30.00

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 08/05/97
BATCH LI03 MOBILE SAMPLES

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	STOP
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 14 Sb 200.9

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	3
LAMP CURRENT (mA)	7
SLIT WIDTH (nm)	0.2
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	217.6
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.40

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	25.0	3.0	ALTERNATE	NO
2	300	25.0	3.0	ALTERNATE	NO
3	900	20.0	3.0	ALTERNATE	NO
4	900	5.0	3.0	ALTERNATE	NO
5	800	1.0	0.0	ALTERNATE	NO
6	2500	0.9	0.0	NORMAL	YES
7	2500	2.5	0.0	NORMAL	YES
8	2700	2.0	3.0	NORMAL	NO
9	45	13.3	3.0	NORMAL	NO

SAMPLER PARAMETERS

VOLUMES (µL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	20	5
STANDARD 1	20	0	5
STANDARD 2	20	0	5
STANDARD 3	20	0	5
STANDARD 4	20	0	5
SAMPLE	20	0	5

RECALIBRATION RATE 0

RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO

TEMPERATURE 100

INJECT RATE 7

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	50
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (µL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	0 TO 200
QC SPIKE RATE	4
QC SPIKE VOLUME (µL)	8
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	2.9 ug/L
REQUIRED DETECTION LIMIT	5.0 ug/L

Zeeman Antimony (3005A) SW846

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR	LR
DATE	02/12/97
BATCH	SB01 0210I,0211L

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 19 Sb HCL std. Amm. Nitr.

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	3
LAMP CURRENT (mA)	7
SLIT WIDTH (nm)	0.2
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	217.6
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.40

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	25.0	3.0	NORMAL	NO
2	300	25.0	3.0	NORMAL	NO
3	700	5.0	3.0	NORMAL	NO
4	700	15.0	3.0	NORMAL	NO
5	700	1.0	0.0	NORMAL	NO
6	2300	0.9	0.0	NORMAL	YES
7	2300	1.5	0.0	NORMAL	YES
8	2600	2.0	3.0	NORMAL	NO
9	45	13.3	3.0	NORMAL	NO

Soil
02.12.97
LR

SAMPLER PARAMETERS
VOLUMES (ℓL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	20	3
STANDARD 1	20	0	3
STANDARD 2	20	0	3
STANDARD 3	20	0	3
STANDARD 4	20	0	3
SAMPLE	20	0	3

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 100
INJECT RATE 7

QC PROTOCOL PARAMETERS

Working Standard Lot # AT-3005

Date Prepared 02.12.97

Analyst LR

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (ℓL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	39 TO 120
QC SPIKE RATE	0
QC SPIKE VOLUME (ℓL)	8
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	1.3 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.0		0.003	0.005	0.001
QC				12Feb97	08:15
				CONCENTRATION LESS THAN IDL	
STANDARD 1	5.0	0.9	0.032	0.032	0.032
QC				12Feb97	08:20
STANDARD 2	10.0	1.9	0.047	0.046	0.047
QC				12Feb97	08:25
STANDARD 3	50.0	5.9	0.217	0.208	0.226
QC				12Feb97	08:30
TANDARD 4	100.0	0.5	0.364	0.365	0.363
.C				12Feb97	08:35

Zeeman — Antimony (HNO3 only) SW846/200 Series 02-27 MM

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR MM
DATE 02/27/97
BATCH SB05 0225E MDL'S

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 18 Sb HNO3

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	3
LAMP CURRENT (mA)	7
SLIT WIDTH (nm)	0.2
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	217.6
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.40

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	25.0	3.0	NORMAL	NO
2	300	25.0	3.0	NORMAL	NO
3	700	20.0	3.0	NORMAL	NO
4	700	5.0	3.0	NORMAL	NO
5	700	1.0	0.0	NORMAL	NO
6	2300	0.9	0.0	NORMAL	YES
7	2300	2.0	0.0	NORMAL	YES
8	2700	2.0	3.0	NORMAL	NO
9	45	13.3	3.0	NORMAL	NO

02-27-97
MM

SAMPLER PARAMETERS
VOLUMES (fL)

	SOLUTION	BLANK	MODIFIER
JK	--	20	5
STANDARD 1	20	0	5
STANDARD 2	20	0	5
STANDARD 3	20	0	5
STANDARD 4	20	0	5
SAMPLE	20	0	5

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 100
INJECT RATE 7

QC PROTOCOL PARAMETERS

Working Standard Lot # LT-Z
Date Prepared 02. 27. 97
Analyst MM

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (fL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	39 TO 111
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	8
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	1.3 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.0		0.001	-0.001	0.002
QC				27Feb97	15:55
				CONCENTRATION LESS THAN IDL	
STANDARD 1	5.0	8.0	0.025	0.026	0.023
QC				27Feb97	16:01
STANDARD 2	10.0	6.6	0.044	0.042	0.046
QC				27Feb97	16:06
STANDARD 3	50.0	1.6	0.208	0.206	0.211
QC				27Feb97	16:11
STANDARD 4	100.0	0.1	0.353	0.353	0.353
				27Feb97	16:16

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 08/05/97
BATCH LI03 MOBILE SAMPLES

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	STOP
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 4 Tl 200.9

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	6
LAMP CURRENT (mA)	10
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	276.8
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	0.55

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	10.0	3.0	ALTERNATE	NO
2	250	10.0	3.0	ALTERNATE	NO
3	950	20.0	3.0	ALTERNATE	NO
4	950	10.0	3.0	ALTERNATE	NO
5	950	1.0	0.0	ALTERNATE	NO
6	2500	0.9	0.0	NORMAL	YES
7	2500	2.0	0.0	NORMAL	YES
8	2600	2.0	3.0	NORMAL	NO
9	40	12.8	3.0	NORMAL	NO

SAMPLER PARAMETERS

VOLUMES (ℓL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	15	5
STANDARD 1	15	0	5
STANDARD 2	15	0	5
STANDARD 3	15	0	5
STANDARD 4	15	0	5
SAMPLE	15	0	5

RECALIBRATION RATE 0

RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO

TEMPERATURE 100

INJECT RATE 7

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	50
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (ℓL)	15
QC STANDARD CONCENTRATION	50.00 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	1
QC SPIKE VOLUME (ℓL)	6
QC SPIKE CONCENTRATION	50.00 ug/L
MATRIX SPIKE CONCENTRATION	50.00 ug/L
INSTRUMENT DETECTION LIMIT	1.23 ug/L
REQUIRED DETECTION LIMIT	2.00 ug/L

Zeeman Tl SW846/200 series

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR MM
DATE 01/24/97
BATCH TL04 0122F RERUNS

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 5 Tl AMM.PHOS.

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	6
LAMP CURRENT (mA)	6
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	276.8
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.00

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	250	20.0	3.0	NORMAL	NO
2	950	20.0	3.0	NORMAL	NO
3	950	10.0	3.0	NORMAL	NO
4	950	1.0	3.0	NORMAL	NO
5	2400	0.9	0.0	NORMAL	YES
6	2400	3.0	0.0	NORMAL	YES
7	2500	2.0	3.0	NORMAL	YES
8	40	12.3	3.0	NORMAL	NO

SAMPLER PARAMETERS
VOLUMES (fL)

	SOLUTION	BLANK	MODIFIER
LANK	--	15	5
STANDARD 1	15	0	5
STANDARD 2	15	0	5
STANDARD 3	15	0	5
STANDARD 4	15	0	5
SAMPLE	15	0	5

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 100
INJECT RATE 7

QC PROTOCOL PARAMETERS

Working Standard Lot # NS
Date Prepared 01.24.97
Analyst MM

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (fL)	15
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	6
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	0.7 ug/L
REQUIRED DETECTION LIMIT	2.0 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.0		0.001	0.001	0.001
QC				24Jan97	13:47
				CONCENTRATION LESS THAN IDL	
STANDARD 1	3.0	8.2	0.009	0.009	0.010
QC				24Jan97	13:51
STANDARD 2	10.0	0.7	0.028	0.027	0.028
QC				24Jan97	13:55
STANDARD 3	50.0	1.9	0.126	0.127	0.124
QC				24Jan97	14:00
STANDARD 4	100.0	2.2	0.213	0.210	0.216
				24Jan97	14:04

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 08/05/97
BATCH LI03 MOBILE SAMPLES

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	STOP
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 12 Pb 200.9

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	4
LAMP CURRENT (mA)	5
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	283.3
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	1
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.40

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	20.0	3.0	ALTERNATE	NO
2	250	10.0	3.0	ALTERNATE	NO
3	250	2.0	3.0	ALTERNATE	NO
4	900	5.0	3.0	ALTERNATE	NO
5	900	20.0	3.0	ALTERNATE	NO
6	900	1.0	0.0	ALTERNATE	NO
7	2400	0.8	0.0	ALTERNATE	YES
8	2400	2.0	0.0	ALTERNATE	YES
9	2400	2.5	3.0	NORMAL	NO
10	45	12.3	3.0	NORMAL	NO

SAMPLER PARAMETERS

VOLUMES (ƒL)

	SOLUTION	BLANK	MODIFIER
ANK	--	10	3
STANDARD 1	10	0	3
STANDARD 2	10	0	3
STANDARD 3	10	0	3
STANDARD 4	10	0	3
SAMPLE	10	0	3

RECALIBRATION RATE 0

RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO

TEMPERATURE 100

INJECT RATE 5

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	50
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (ƒL)	10
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (ƒL)	4
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	0.2 ug/L
REQUIRED DETECTION LIMIT	5.0 ug/L

Zeeman Pb SW846/200 series

Pb01
12.05.9
LR

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 12/05/96
BATCH PB01,TL01 IDL'S

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 1 Pb CLP AMM.PHOS.

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	4
LAMP CURRENT (mA)	5
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	NORMAL
WAVELENGTH (nm)	283.3
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.40

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	150	20.0	3.0	NORMAL	NO
2	250	10.0	3.0	NORMAL	NO
3	250	2.0	3.0	NORMAL	NO
4	600	5.0	3.0	NORMAL	NO
5	600	20.0	3.0	NORMAL	NO
6	900	1.0	3.0	NORMAL	NO
7	900	2.0	0.0	NORMAL	NO
8	2100	0.8	0.0	NORMAL	YES
9	2100	2.0	0.0	NORMAL	YES
10	2400	2.5	3.0	NORMAL	NO
11	45	12.3	3.0	NORMAL	NO

12.05.96
LR

SAMPLER PARAMETERS
VOLUMES (fL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	20	3
STANDARD 1	20	0	3
STANDARD 2	20	0	3
STANDARD 3	20	0	3
STANDARD 4	20	0	3
SAMPLE	20	0	3

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 100
INJECT RATE 5

QC PROTOCOL PARAMETERS

Working Standard Lot # JR
Date Prepared 12.05.96
Analyst LR

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (fL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	8
QC SPIKE CONCENTRATION	50.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	0.2 ug/L
REQUIRED DETECTION LIMIT	5.0 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.0		0.003	0.003	0.002
QC				05Dec96	16:10
				CONCENTRATION LESS THAN IDL	
STANDARD 1	3.0	5.5	0.017	0.016	0.018
QC				05Dec96	16:14
STANDARD 2	10.0	3.5	0.063	0.061	0.064
QC				05Dec96	16:19
STANDARD 3	50.0	0.7	0.290	0.288	0.291
QC				05Dec96	16:23
STANDARD 4	100.0	0.2	0.528	0.527	0.529
QC				05Dec96	16:28

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 02/14/97
BATCH CR02,CU02 MDL'S

Zeeman Cr SW846/200 series

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 16 Cr NO MODIFIER

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	8
LAMP CURRENT (mA)	7
SLIT WIDTH (nm)	0.2
SLIT HEIGHT	REDUCED
WAVELENGTH (nm)	357.9
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.50

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	95	30.0	3.0	NORMAL	NO
2	120	20.0	3.0	NORMAL	NO
3	500	5.0	3.0	NORMAL	NO
4	500	2.0	3.0	NORMAL	NO
5	1000	5.0	3.0	NORMAL	NO
6	1000	5.0	3.0	NORMAL	NO
7	1000	2.0	0.1	NORMAL	NO
8	2700	1.1	0.0	NORMAL	YES
9	2700	2.0	0.0	NORMAL	YES
10	2700	3.0	3.0	NORMAL	NO
11	80	13.1	3.0	NORMAL	NO

SAMPLER PARAMETERS

VOLUMES (fL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	6	
STANDARD 1	6	0	
STANDARD 2	6	0	
STANDARD 3	6	0	
STANDARD 4	6	0	
SAMPLE	6	0	

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 95
INJECT RATE 7

QC PROTOCOL PARAMETERS

Working Standard Lot # CT-2

Date Prepared 02-14-97

Analyst LN

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (fL)	6
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	6
QC SPIKE CONCENTRATION	20.0 ug/L
MATRIX SPIKE CONCENTRATION	50.0 ug/L
INSTRUMENT DETECTION LIMIT	0.3 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK QC	0.0		0.034	0.042	0.025
				14Feb97	13:03
				CONCENTRATION LESS THAN IDL	
BLANK QC	0.0		-0.020	-0.019	-0.021
				14Feb97	13:09
				CONCENTRATION LESS THAN IDL	
STANDARD 1 QC	5.0	0.3	0.052	0.052	0.052
				14Feb97	13:13
STANDARD 2 QC	10.0	2.2	0.103	0.104	0.101
				14Feb97	13:18
STANDARD 3	50.0	3.2	0.498	0.486	0.509
				14Feb97	13:22
STANDARD 4 QC	100.0	0.3	0.924	0.922	0.926
				14Feb97	13:26

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR LR
DATE 02/27/97
BATCH LI05 70676 AUDIT,MDL'S

Zeeman Li "SW846-like"

QC PROTOCOL PARAMETERS

IF THERE IS AN ERROR	RESLOPE AND REPEAT
QC SPIKE POSITION	52
RECOVERY LIMITS (%)	85 TO 115
RECOVERY MINIMUM LIMIT (%)	40
CORRELATION COEFFICIENT (r)	0.995
OVERRANGE VOLUME REDUCTION	1
REPLICATE RSD LIMIT (%)	20.0

PROGRAM 23 Li 5% H2/ARGON

INSTRUMENT MODE	ABSORBANCE
CALIBRATION MODE	CONCENTRATION
MEASUREMENT MODE	PEAK AREA
LAMP POSITION	1
LAMP CURRENT (mA)	5
SLIT WIDTH (nm)	0.5
SLIT HEIGHT	REDUCED
WAVELENGTH (nm)	670.8
SAMPLE INTRODUCTION	SAMPLER PREMIXED
TIME CONSTANT	0.05
MEASUREMENT TIME (sec)	1.0
REPLICATES	2
BACKGROUND CORRECTION	ON
MAXIMUM ABSORBANCE	1.40

FURNACE PARAMETERS

STEP NO.	TEMPERATURE (C)	TIME (sec)	GAS FLOW (L/min)	GAS TYPE	READ COMMAND
1	90	5.0	3.0	ALTERNATE	NO
2	120	5.0	3.0	ALTERNATE	NO
3	200	20.0	3.0	ALTERNATE	NO
4	600	5.0	3.0	ALTERNATE	NO
5	600	10.0	3.0	ALTERNATE	NO
6	600	1.0	2.0	ALTERNATE	NO
7	2600	1.1	2.0	ALTERNATE	YES
8	2600	1.2	2.0	ALTERNATE	YES
9	2800	2.0	3.0	NORMAL	NO
10	45	13.8	3.0	NORMAL	NO

LR

SAMPLER PARAMETERS
VOLUMES (µL)

	SOLUTION	BLANK	MODIFIER
F K	--	8	12
DARD 1	8	0	12
STANDARD 2	8	0	12
STANDARD 3	8	0	12
STANDARD 4	8	0	12
SAMPLE	8	0	12

RECALIBRATION RATE 0
RESLOPE RATE 0

MULTIPLE INJECT NO HOT INJECT YES PRE INJECT NO
TEMPERATURE 80
INJECT RATE 5

QC PROTOCOL PARAMETERS

QC STANDARD RATE	20
QC INITIAL STANDARD POSITION	45
QC INITIAL BLANK POSITION	51
QC CONTINUING STANDARD POSITION	50
QC CONTINUING BLANK POSITION	51
QC STANDARD VOLUME (µL)	8
QC STANDARD CONCENTRATION	25.00 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (µL)	4
QC SPIKE CONCENTRATION	20.00 ug/L
MATRIX SPIKE CONCENTRATION	25.00 ug/L
INSTRUMENT DETECTION LIMIT	0.70 ug/L
REQUIRED DETECTION LIMIT	10.00 ug/L

Working Standard Lot # LT-LI

Date Prepared 02.27.97

Analyst LR

SAMPLE	CONC ug/L	%RSD	MEAN ABS	READINGS	
BLANK	0.00		0.002	0.001	0.002
QC				27Feb97	06:53
				CONCENTRATION LESS THAN IDL	
STANDARD 1	5.00	10.1	0.007	0.007	0.006
QC				27Feb97	06:57
STANDARD 2	10.00	3.6	0.017	0.017	0.018
QC				27Feb97	07:00
STANDARD 3	20.00	0.9	0.037	0.037	0.036
QC				27Feb97	07:05
STANDARD 4	50.00	1.8	0.097	0.095	0.098
QC				27Feb97	07:08

10.3 Samples are analyzed only after the DFTPP criteria and the calibration acceptance criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the DFTPP and a mid-level calibration standard(usually the 80ng standard).

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
DFTPP 50ng on column Pentachlorophenol, benzidine, p,p'-DDT added for column evaluation Clock starts at injection	DFTPP 50ng on column Pentachlorophenol, benzidine, p,p'-DDT added for column evaluation Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification (50ug/mL or 80ug/mL) Optional RL: Standard-low point on cal curve
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

10.3.1 The percent difference or percent drift between the continuing calibration RRF and the average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$\%difference = \left| \frac{RRF - RRFavg}{RRFavg} \right| \otimes 100$$

where
RRF = relative response factor from CCV
RRFavg = average relative response factor from initial calibration curve

$$\%drift = \left| \frac{Ccurve - Ct}{Ct} \right| \otimes 100$$

where
Ccurve = concentration from curve(ug/mL)
Ct = true concentration of CCV

The continuing calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in the SOP summary. The %D (Drift)criteria for CCC and minimum RRF for SPCCs must be met before the analysis of sample extracts can begin.

If the continuing calibration criteria are not met, action must taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the analysis of the continuing calibration standard must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards. If the continuing calibration standard repeatedly fails the CCC and SPCC criteria, the initial calibration curve must be reanalyzed and reevaluated.

The initial calibration must be evaluated for the CCC and SPCC criteria even though the calibration curve may be used for quantitation.

10.3.2 Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.3.3 Add 20-uL of the internal standard mix (2000ng/uL) to each 1-mL aliquot of the sample extract. The concentration of the internal standard in the extract is 40ng/uL.

10.3.4 Mix the contents of the autosampler vial by inverting several times.

10.3.5 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

10.3.6 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract is diluted to final volume of 1.0mL, the dilution factor is 10. (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (2000ug/mL)-Vistd	DF
1000	0	1000	20	1
500	500	1000	10*	2
200	800	1000	16*	5
100	900	1000	18*	10
50	950	1000	19*	20
20	980	1000	20*	50

ssumes dilution of a 1.0mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 40ug/mL using 20ul of a 2000ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 40ug/mL. The following equation can be used to determine the volume of the 2000ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the interanl standard solution:

$$Vistd(uL) = 20uL - \left(\frac{Vext}{Vdil} \otimes 20ul\right)$$

Vistd = volume of 2000ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

11.1.1 Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the daily calibration standard or a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to positively identify a compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{\textit{retention time of the target compound}}{\textit{retention time of the associated internal standard}}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. Ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification..

11.1.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.2.2 The relative intensities of the major ions should agree within +/-30%.

11.1.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.1.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.1.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.1.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult senior analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.1.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculations assume that the same volume is injected for standards and samples.

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{V} \otimes DF$$

where

Cis =	concentration of the internal standard (ug/mL)
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract (mL)
V =	volume of sample extract (L)
DF =	dilution factor

Soils

$$TIC\ (ug/kg, dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Cis =	concentration of the internal standard, ug/mL
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract mL
W =	weight of sample analyzed (kg)
solids =	decimal equivalent of percent solids

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples.

1.2.1 Aqueous Samples

11.2.1.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.2 If a regression curve is used, the concentration is given:

$$concentration(ug/L) = Ccurve \otimes \frac{F}{V} \otimes DF$$

where

C_{curve} =	concentration from curve (ug/mL)
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.3 The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where

F =	final volume of extract (mL)
Fqap =	1.0mL
Vqap =	1.0L
V =	volume of sample extracted
DF =	dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

11.2.2.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

11.2.2.2 If the regression curve is used, the concentration is given:

$$conc(ug/kg,dw) = Ccurve \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ccurve =	concentration from curve(ug/mL)
W =	weight of sample extracted (kg)
F =	final volume of extract (mL)
solids =	(percent solids)/100)
DF =	dilution factor

2.2.3 The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100

The SL CQAP assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

11.3 The concentration of SVOC in the lab control and matrix spike samples is determined as in Section 11.1. The concentration is compared to the theoretical spike concentration and the percent recovery is calculated.

11.3.1 The theoretical or true value for a spiked sample is calculated using the following equation:

$$concentration = \frac{amount\ of\ compound\ added}{volume\ /\ weight\ of\ sample\ extracted}$$

For liquid samples, this equation expands to:

$$concentration(ug/L) = \frac{Cspike \otimes Vspike}{Vsample}$$

where

Cspike =	concentration of the spiking solution (ug/mL)
Vspike =	volume of spiking solution added to the sample (mL)
Vsample =	volume of sample spiked (L)

For soil and solid samples, the equation expands to:

$$concentration(ug/L) = \frac{Cspike \otimes Vspike}{Wsample \otimes solids}$$

where

Cspike =	concentration of the spiking solution (ug/mL)
Vspike =	volume of spiking solution added to the sample (mL)
Wsample =	weight of sample spiked (kg)
solids =	(percent solids)/100

11.3.2 The percent recovery of the target analytes in the matrix spike sample and lab control samples is calculated:

$$\%recovery\ (\%REC) = \frac{(Cms - Cs)}{Ts} x\ 100$$

where

Cms =	concentration of the spiked sample (MS/MSD or LCS/LCSD)
Cs =	concentration of the unspiked sample
Ts =	theoretical concentration of the spike

11.3.3 The matrix spike sample is analyzed in duplicate(MS/MSD). The relative percent difference (%RPD), a measure of precision, is calculated for the MS/MSD pair as follows:

$$\%RPD = \left| \frac{\%REC(MS) - \%REC(MSD)}{(\%REC(MS) + (\%REC(MSD))/2} \right| x\ 100$$

The absolute value of the %RPD is evaluated and reported; that is, only positive values are reported. If the MS/MSD cannot be performed, LCS/LCSD is analyzed and evaluated.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. Note that the method blank for liquid samples and low level soils is clock-specific and that the method blank for medium level soil samples is batch-specific

SL-SOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. The attached SOP summary and Table 13.1 in the SL Corporate QA Plan provide guidance for evaluating sample data.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration are the preparation and analysis of spiked reagent water. The criteria for IDOC accuracy and precision are given in the SW-846 Method 8270.

12.3 Method Detection Limit

The method detection limit is determined by each lab annually in accordance with SL SOP CA90:*Procedure for Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. See Section 10 of the current SL Quality Assurance Plan.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time.

15.0 REFERENCES

15.1 *Savannah Laboratories' Comprehensive Quality Assurance Plan* and Savannah Laboratories' *Corporate Quality Assurance Plan*, current revisions.

15.2 Method 8270C:*Test Methods for Evaluating Solid Wastes, Third Edition, SW-846.* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

8270 SOP SUMMARY

OLD TIMES

MATRIX	Preservative/ storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
DFTPP 50ng on column Pentachlorophenol, benzidine, p,p'-DDT added for column evaluation Clock starts at injection	DFTPP 50ng on column Pentachlorophenol, benzidine, p,p'-DDT added for column evaluation Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification (50ug/mL or 80ug/mL) RL Standard (lowest point on calibration curve if required by client or state-specific QAP)
amples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

DFTPP CRITERIA

m/z	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass443
442	>40% of mass 198
443	17-23% of mass 442

ANALYTICAL BATCH

SEE SL SOP AN02.

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Phenol, 1,4-Dichlorobenzene, 2-Nitrophenol, 2,4-Dichlorophenol, Hexachlorobutadiene, 4-Chloro-3-methylphenol, 2,4.6-Trichlorophenol, Acenapthene, N-Nitrosodiphenylamine, Pentachlorophenol, Fluoranthene, Di-n-octylphthalate, Benzo(a) pyrene

System Performance Check Compounds-SPCC
N-Nitrosodi-n-propylamine, Hexachlorocyclopentadiene,
2,4-Dinitrophenol, 4-Nitrophenol

Initial Calibration	Continuing Calibration*
CCC: <= 30% RSD	CCC: <= 20% difference from initial calibration
SPCC: RRFavg >= 0.050	SPCC: RRF>= 0.050

*If CCC and/or SPCC do not meet the stated criteria, all targets that are reported must meet the CCC criteria.

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

The results for all target compounds are evaluated for linearity. If the %RSD is less than 15%, the calibration is assumed linear through the origin and the average response factor can be used for quantitation. If the average response factor for the target exceeds 15% (including any CCC), the analyst must use the calibration curve option.

NOTE: The lab has the option of using a calibration curve for all analytes. This is the recommended course of action-especially for data systems that cannot automatically calculate the relative response factors or when choosing calibration options compound by compound will be time consuming.

A linear, quadratic, or higher order regression fit may be used to define the concentration/response relationship. If the correlation coefficient of the linear regression curve or the coefficient of determination of a higher order fit is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range. The minimum number of calibration standards required for a regression curve are given in the following table:

Type of curve	Minimum Number of Calibration Points
Linear (first order)	5
Quadratic (second order)	6
Polynomial(third)	7

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 50ng Pentachlorophenol - 50ng Benzidine – 50ng p,p'-DDT 50ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
		Pentachlorophenol and benzidine - present at usual response with no peak tailing visible p,p'-DDT - %breakdown <20%	-Reanalyze -Perform injector port maintenance and reanalyze -Cut more than usual length of column and reanalyze -Replace column
Initial Calibration	After Tune Check and when calibration verification standard fails acceptance criteria. All initial calibration standards	CCC: %RSD < 30% SPCC: RRFavg > 0.050 Used cal curve for quantitation if %RSD for any target compound exceeds 15%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples	CCC: %Difference <= 20% Or %Drift <= 20% SPCC: RRF >= 0.050	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be 50% to +200% of previous midlevel standard Areas in samples should be evaluated for gross error . Consult superior	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available

QC Item	Frequency	Acceptance Criteria	Corrective Action
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within Section 5 QAP limits; one acid/one base may be outside of criteria	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1
Lab Control Standard (LCS) - QAP subset	See AN02	All spiked targets within the accuracy criteria in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See AN02	All targets within the accuracy and precision criteria in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1
RL Standard (reporting limit)	Daily (optional)-lowest point on calibration curve if required by client or state-specific QAP	Detected at reasonable sensitivity	-Evaluate integrations and spectra; -Reanalyze -Prepare new standard and reanalyze
Initial Demonstration of Capability (IDOC)	Per analyst	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	See CA90	Evaluate according to SL SOP CA90	Evaluate according to SL SOP CA90

SM05 APPENDIX- EXAMPLE QUANTITATION REPORT

SAVANNAH LABORATORIES

COMPOUND LISTING

Method file : /chem/SM/MSF5970.i/f-b8270-m:m
Quant Method : ISTD
Last Update : 04-Dec-96 14:43:5
Data Type : MS DATA

Target Version : 3.12
Number of Cpnds : 91

Global Integrator : HP RTE

Chromat Events	Values
Initial:Thresh Units	0.000000
Initial:Area Thresh	100.000000
Initial:Max Peaks	100.000000
Initial:Bunching	4.000000
Initial:Smoothing	0.000000
Initial:Start Thresh	0.155000
Initial:Stop Thresh	0.000000
Initial:Baseline Reset	5.000000
Initial:Set Valley	100.000000

	Compound	RT	RT Window	RF	Mass
*	1 1,4-Dichlorobenzene-d4	8.044	7.561-8.527	863.105	152.10
		8.044	7.561-8.527	0.000	150.10
		8.044	7.561-8.527	0.000	115.10
	2 1,4-Dioxane	1.946	1.463-2.429	0.400	88.00
		1.946	1.463-2.429	0.000	58.00
		1.946	1.463-2.429	0.000	45.00
	3 Pyridine	2.528	2.045-3.011	1.481	79.00
		2.528	2.045-3.011	0.000	52.00
		2.528	2.045-3.011	0.000	51.00
	4 N-Nitrosodimethylamine	2.609	2.126-3.092	1.054	42.00
		2.609	2.126-3.092	0.000	74.00
$	5 2-Fluorophenol	5.194	4.711-5.677	1.373	112.00
		5.194	4.711-5.677	0.000	64.00
$	6 Phenol-d5	7.409	6.926-7.892	1.801	99.10
		7.409	6.926-7.892	0.000	71.10
	7 Aniline	7.349	6.866-7.832	1.830	93.10
		7.349	6.866-7.832	0.000	66.00
	8 Phenol	7.442	6.959-7.924	2.009	94.10
		7.442	6.959-7.924	0.000	66.10
	9 Bis(2-chloroethyl)ether	7.561	7.078-8.044	1.716	93.00
		7.561	7.078-8.044	0.000	63.00
		7.561	7.078-8.044	0.000	95.00

* = internal standard
$ = surrogate WR 12/23/97

SAVANNAH LABORATORIES

COMPOUND LISTING

Method file : /chem/SM/MSF5970.i/f-b8270-m.m

Compound	RT	RT Window	RF	Mass
10 2-Chlorophenol	7.594	7.111-8.076	1.489	128.10
	7.594	7.111-8.076	0.000	130.10
	7.594	7.111-8.076	0.000	64.00
11 1,3-Dichlorobenzene	7.898	7.415-8.380	1.523	146.00
	7.898	7.415-8.380	0.000	148.00
	7.898	7.415-8.380	0.000	111.00
12 1,4-Dichlorobenzene	8.088	7.605-8.570	1.564	146.00
	8.088	7.605-8.570	0.000	148.00
	8.088	7.605-8.570	0.000	111.00
13 Benzyl Alcohol	8.490	8.007-8.973	0.976	108.10
	8.490	8.007-8.973	0.000	79.10
14 1,2-Dichlorobenzene	8.419	7.936-8.901	1.465	146.00
	8.419	7.936-8.901	0.000	148.00
	8.419	7.936-8.901	0.000	111.00
15 2-Methylphenol	8.815	8.332-9.297	1.255	107.00
	8.815	8.332-9.297	0.000	108.00
	8.815	8.332-9.297	0.000	77.00
	8.815	8.332-9.297	0.000	79.00
16 bis(2-Chloroisopropyl)ethe	8.799	8.316-9.282	2.025	45.00
	8.799	8.316-9.282	0.000	77.00
	8.799	8.316-9.282	0.000	121.00
17 N-Nitroso-di-n-propylamine	9.152	8.669-9.635	1.325	70.00
	9.152	8.669-9.635	0.000	130.00
	9.152	8.669-9.635	0.000	101.00
	9.152	8.669-9.635	0.000	42.00
18 3&4-Methylphenol	9.217	8.734-9.699	1.648	107.10
	9.217	8.734-9.699	0.000	108.10
	9.217	8.734-9.699	0.000	77.10
19 Hexachloroethane	9.201	8.718-9.683	0.762	116.80
	9.201	8.718-9.683	0.000	200.80
	9.201	8.718-9.683	0.000	198.80
* 20 Naphthalene-d8	11.101	10.435-11.767	3374	136.00
	11.101	10.435-11.767	0.000	68.00
$ 21 Nitrobenzene-d5	9.402	8.736-10.068	0.520	82.10
	9.402	8.736-10.068	0.000	128.10
	9.402	8.736-10.068	0.000	54.00
22 Nitrobenzene	9.456	8.790-10.122	0.541	77.10
	9.456	8.790-10.122	0.000	123.00
	9.456	8.790-10.122	0.000	65.00
23 Isophorone	10.085	9.419-10.751	0.960	82.00
	10.085	9.419-10.751	0.000	138.00
	10.085	9.419-10.751	0.000	95.00

SAVANNAH LABORATORIES

COMPOUND LISTING

Method file : /chem/SM/MSF5970.i/f-b8270-m.m

Compound	RT	RT Window	RF	Mass
24 2-Nitrophenol	10.210	9.544-10.876	0.185	139.00
	10.210	9.544-10.876	0.000	109.00
	10.210	9.544-10.876	0.000	65.00
25 2,4-Dimethyphenol	10.476	9.810-11.142	0.000	107.00
	10.476	9.810-11.142	0.301	122.10
	10.476	9.810-11.142	0.000	121.00
26 Bis(2-chloroethoxy)methane	10.677	10.011-11.343	0.595	93.00
	10.677	10.011-11.343	0.000	123.00
	10.677	10.011-11.343	0.000	95.00
27 Benzoic Acid	10.981	10.315-11.647	0.174	122.00
	10.981	10.315-11.647	0.000	77.00
	10.981	10.315-11.647	0.000	105.00
28 2,4-Dichlorophenol	10.861	10.195-11.527	0.289	162.00
	10.861	10.195-11.527	0.000	164.00
	10.861	10.195-11.527	0.000	98.00
29 1,2,4-Trichlorobenzene	10.987	10.320-11.653	0.315	180.00
	10.987	10.320-11.653	0.000	182.00
	10.987	10.320-11.653	0.000	145.00
30 Naphthalene	11.161	10.495-11.827	1.061	128.00
	11.161	10.495-11.827	0.000	129.00
	11.161	10.495-11.827	0.000	127.00
31 4-Chloroaniline	11.395	10.728-12.060	0.256	127.10
	11.395	10.728-12.060	0.000	129.10
	11.395	10.728-12.060	0.000	65.00
	11.395	10.728-12.060	0.000	92.00
32 Hexachlorobutadiene	11.504	10.837-12.169	0.180	224.80
	11.504	10.837-12.169	0.000	222.80
	11.504	10.837-12.169	0.000	226.80
33 N-Nitroso-di-n-butylamine	12.297	11.639-12.972	0.317	84.00
	12.297	11.639-12.972	0.000	57.00
	12.297	11.639-12.972	0.000	41.00
34 4-Chloro-3-Methylphenol	12.719	12.053-13.385	0.393	106.80
	12.719	12.053-13.385	0.000	143.80
	12.719	12.053-13.385	0.000	141.80
35 2-Methylnaphthalene	12.904	12.237-13.569	0.628	142.00
	12.904	12.237-13.569	0.000	141.00
36 1-Methylnaphthalene	13.143	12.476-13.809	0.582	142.00
	13.143	12.476-13.809	0.000	141.00
* 37 Acenaphthene-d10	15.507	14.577-16.437	1712	164.00
	15.507	14.577-16.437	0.000	162.00
	15.507	14.577-16.437	0.000	160.00

SAVANNAH LABORATORIES

COMPOUND LISTING

Method file : /chem/SM/MSF5970.i/f-b8270-m.m

	Compound	RT	RT Window	RF	Mass
	38 Hexachlorocyclopentadiene	13.301	12.370-14.231	0.355	236.80
		13.301	12.370-14.231	0.000	271.80
	39 2,4,6-Trichlorophenol	13.675	12.744-14.605	0.433	196.00
		13.675	12.744-14.605	0.000	198.00
		13.675	12.744-14.605	0.000	200.00
	40 2,4,5-Trichlorophenol	13.789	12.858-14.719	0.413	196.00
		13.789	12.858-14.719	0.000	198.00
		13.789	12.858-14.719	0.000	200.00
M	41 Trichlorophenols	13.675	13.192-14.158	0.000	100.00
$	42 2-Fluorobiphenyl	13.893	12.963-14.823	1.391	172.00
		13.893	12.963-14.823	0.000	171.00
		13.893	12.963-14.823	0.000	170.00
	43 2-Chloronaphthalene	14.121	13.191-15.052	1.262	162.10
		14.121	13.191-15.052	0.000	164.10
		14.121	13.191-15.052	0.000	127.10
	44 2-Nitroaniline	14.474	13.543-15.404	0.626	65.10
		14.474	13.543-15.404	0.000	92.10
		14.474	13.543-15.404	0.000	138.10
	45 Dimethylphthalate	15.016	14.086-15.947	1.567	163.00
		15.016	14.086-15.947	0.000	194.00
		15.016	14.086-15.947	0.000	164.00
	46 2,6-Dinitrotoluene	15.132	14.202-16.063	0.342	165.00
		15.132	14.202-16.063	0.000	89.00
		15.132	14.202-16.063	0.000	63.00
	47 Acenaphthylene	15.149	14.218-16.079	1.764	152.10
		15.149	14.218-16.079	0.000	151.10
		15.149	14.218-16.079	0.000	153.10
	48 3-Nitroaniline	15.485	14.554-16.415	0.105	138.10
		15.485	14.554-16.415	0.000	108.00
		15.485	14.554-16.415	0.000	92.10
	49 Acenaphthene	15.600	14.669-16.530	1.109	154.10
		15.600	14.669-16.530	0.000	153.10
		15.600	14.669-16.530	0.000	152.10
	50 2,4-Dinitrophenol	15.795	14.864-16.725	0.133	184.00
		15.795	14.864-16.725	0.000	107.00
		15.795	14.864-16.725	0.000	154.00
	51 4-Nitrophenol	16.144	15.213-17.074	0.273	65.00
		16.144	15.213-17.074	0.000	109.00
		16.144	15.213-17.074	0.000	139.00
	52 Dibenzofuran	16.045	15.114-16.975	1.699	168.10
		16.045	15.114-16.975	0.000	139.10

SAVANNAH LABORATORIES

COMPOUND LISTING

Method file : /chem/SM/MSF5970.i/f-b8270-m.m

	Compound	RT	RT Window	RF	Mass
	53 2,4-Dinitrotoluene	16.133	15.202-17.063	0.453	165.10
		16.133	15.202-17.063	0.000	89.00
	54 2,3,4,6-Tetrachlorophenol	16.410	15.480-17.340	0.342	231.80
		16.410	15.480-17.340	0.000	229.80
		16.410	15.480-17.340	0.000	130.80
M	55 Tetrachlorophenols	1.000	0.980-1.020	0.000	100.00
	56 Diethylphthalate	16.828	15.898-17.759	1.702	149.10
		16.828	15.898-17.759	0.000	177.10
	57 Fluorene	16.916	15.985-17.846	1.013	166.10
		16.916	15.985-17.846	0.000	165.10
		16.916	15.985-17.846	0.000	167.10
	58 4-Chlorophenyl-phenylether	16.987	16.056-17.917	0.469	203.80
		16.987	16.056-17.917	0.000	140.80
		16.987	16.056-17.917	0.000	205.80
	59 4-Nitroaniline	17.123	16.192-18.053	0.129	138.10
		17.123	16.192-18.053	0.000	108.00
		17.123	16.192-18.053	0.000	92.00
$	60 2,4,6-Tribromophenol	17.526	16.595-18.456	0.202	330.00
		17.526	16.595-18.456	0.000	332.00
		17.526	16.595-18.456	0.000	141.00
*	61 Phenanthrene-d10	19.271	18.115-20.427	2525	188.00
		19.271	18.115-20.427	0.000	94.00
		19.271	18.115-20.427	0.000	189.00
	62 4,6-Dinitro-2-methylphenol	17.145	15.989-18.301	0.134	198.10
		17.145	15.989-18.301	0.000	105.10
	63 Diphenylamine	17.309	16.154-18.467	0.768	169.00
		17.309	16.154-18.467	0.000	168.00
		17.309	16.154-18.467	0.000	167.00
	64 N-Nitrosodiphenylamine	17.319	16.163-18.475	0.421	169.00
		17.319	16.163-18.475	0.000	168.00
		17.319	16.163-18.475	0.000	167.00
	65 1,2-Diphenylhydrazine	17.390	16.234-18.546	1.477	77.00
		17.390	16.234-18.546	0.000	105.00
		17.390	16.234-18.546	0.000	182.00
	66 4-Bromophenyl-phenylether	18.221	17.065-19.377	0.228	248.00
		18.221	17.065-19.377	0.000	250.00
		18.221	17.065-19.377	0.000	141.00
	67 Hexachlorobenzene	18.281	17.125-19.438	0.327	283.80
		18.281	17.125-19.438	0.000	141.80
		18.281	17.125-19.438	0.000	248.80

SAVANNAH LABORATORIES

COMPOUND LISTING

Method file : /chem/SM/MSF5970.i/f-b8270-m.m

Compound	RT	RT Window	RF	Mass
68 Pentachlorophenol	18.846	17.690-20.002	0.205	265.80
	18.846	17.690-20.002	0.000	263.80
	18.846	17.690-20.002	0.000	267.80
69 Phenanthrene	19.342	18.186-20.498	1.209	178.00
	19.342	18.186-20.498	0.000	176.00
70 Anthracene	19.473	18.316-20.629	1.109	178.00
	19.473	18.316-20.629	0.000	176.00
	19.473	18.316-20.629	0.000	179.00
71 Carbazole	20.016	18.860-21.172	0.488	167.00
72 Di-n-Butylphthalate	20.988	19.832-22.145	1.935	149.10
	20.988	19.832-22.145	0.000	150.10
73 Fluoranthene	22.357	21.201-23.513	1.183	202.10
	22.357	21.201-23.513	0.000	101.00
	22.357	21.201-23.513	0.000	203.00
74 Benzidine	25.515	24.359-26.671	0.054	184.00
	25.515	24.359-26.671	0.000	92.00
* 75 Chrysene-d12	26.017	24.456-27.578	2460	240.00
	26.017	24.456-27.578	0.000	236.00
	26.017	24.456-27.578	0.000	120.00
76 Pyrene	22.922	21.360-24.482	1.537	202.10
	22.922	21.360-24.482	0.000	200.10
	22.922	21.360-24.482	0.000	203.10
$ 77 Terphenyl-d14	23.433	21.872-24.994	1.005	244.10
	23.433	21.872-24.994	0.000	122.10
	23.433	21.872-24.994	0.000	212.10
78 Butylbenzylphthalate	24.776	23.215-26.337	1.066	149.10
	24.776	23.215-26.337	0.000	91.10
	24.776	23.215-26.337	0.000	206.00
79 3,3'-Dichlorobenzidine	26.033	24.472-27.594	0.272	252.00
	26.033	24.472-27.594	0.000	254.00
	26.033	24.472-27.594	0.000	126.00
80 Benzo(a)Anthracene	25.995	24.434-27.556	1.233	228.00
	25.995	24.434-27.556	0.000	229.00
	25.995	24.434-27.556	0.000	226.00
	25.995	24.434-27.556	0.000	114.10
81 bis(2-ethylhexyl)Phthalate	26.284	24.723-27.845	1.406	149.00
	26.284	24.723-27.845	0.000	167.00
	26.284	24.723-27.845	0.000	279.00
82 Chrysene	26.088	24.527-27.649	1.263	228.00
	26.088	24.527-27.649	0.000	226.00
	26.088	24.527-27.649	0.000	229.00
	26.088	24.527-27.649	0.000	114.00

SAVANNAH LABORATORIES

COMPOUND LISTING

Method file : /chem/SM/MSF5970.i/f-b8270-m.m

	Compound	RT	RT Window	RF	Mass
*	83 Perylene-d12	29.413	27.648-31.178	2305	264.10
		29.413	27.648-31.178	0.000	265.00
		29.413	27.648-31.178	0.000	260.00
	84 Di-n-octylphthalate	27.861	26.096-29.626	2.440	149.10
		27.861	26.096-29.626	0.000	167.10
		27.861	26.096-29.626	0.000	43.10
	85 Benzo(b)fluoranthene	28.544	26.779-30.309	1.320	252.00
		28.544	26.779-30.309	0.000	253.00
		28.544	26.779-30.309	0.000	125.00
	86 Benzo(k)fluoranthene	28.615	26.850-30.380	1.173	252.00
		28.615	26.850-30.380	0.000	253.00
		28.615	26.850-30.380	0.000	125.00
M	87 Benzo(b,k)fluoranthene	28.544	0.980-1.020	0.000	100.00
	88 Benzo(a)pyrene	29.287	27.522-31.052	1.090	252.00
		29.287	27.522-31.052	0.000	125.00
		29.287	27.522-31.052	0.000	253.00
	89 Indeno(1,2,3-cd)pyrene	32.544	30.779-34.309	0.950	276.00
		32.544	30.779-34.309	0.000	138.10
		32.544	30.779-34.309	0.000	277.00
	90 Dibenzo(a,h)anthracene	32.648	30.883-34.413	0.773	278.00
		32.648	30.883-34.413	0.000	139.00
		32.648	30.883-34.413	0.000	279.00
	91 Benzo(g,h,i)perylene	33.499	31.734-35.264	0.950	276.00
		33.499	31.734-35.264	0.000	138.00
		33.499	31.734-35.264	0.000	277.00

CHANGE -IN-PROGRESS ATTACHMENT

SOP Document No: VG15:07.15.98:5
SOP Description: VOLATILE PETROLEUM PRODUCTS AND VOLATILE HYDROCARBONS AS GRO (MODIFIED 8015B)

Approval Signature: R. Wayne Robbins	
Title: Corporate QA Manager	Date: 9/4/98

The following revisions or additions have been made to the referenced SOP.

12.4 Contamination by carryover can occur whenever high concentration samples and low concentration samples are analyzed sequentially. Where practical, the high concentration samples should be followed by a blank to check for cross-contamination. If the targets found in the highly concentrated sample are found in subsequent samples, the analyst must verify that the port and P/T system are not contributing contamination to the subsequent samples. If the target compound(s) are <u>not</u> present in subsequent samples, the analysis of a blank is not required but may be a prudent preventative measure.

Approval Signature: R. Wayne Robbins	
Title: Corporate QA Manager	Date: July 17, 1998

VOLATILE PETROLEUM PRODUCTS AND VOLATILE HYDROCARBONS AS GRO (MODIFIED 8015B)

1.0 SCOPE AND APPLICATION

1.1 This procedure can be used to identify and quantify volatile petroleum-based solvent mixtures in aqueous samples, soils and sediments, and waste samples by purge and trap/gas chromatography. Gasoline is the primary target compounds but other petroleum products can be determined if the product is provided to the lab. This procedure also contains the procedures for determining and reporting the concentration of volatile hydrocarbons in the gasoline range, commonly referred to as GRO(gasoline range organics). BTEX can be measured concurrently if the PID/FID detector configuration is used. Example chromatograms are given in the appendix to this SOP.

1.2 The reporting limit(RL), the method detection limit(MDL), and the accuracy and precision limits are listed in Section 5 of the Savannah Laboratories' quality assurance plans.

2.0 SUMMARY OF METHOD

2.1 Volatile petroleum products and other volatile materials are purged from the sample matrix by helium. Aqueous samples are purged at ambient conditions and solids are purged at 40C. The volatile constituents are trapped on an adsorbent material and thermally desorbed on to a temperature programmable GC equipped with a flame ionization detector. If aromatics (BTEX) are to be determined concurrently, the GC is equipped with an FID and photoionization detector (PID). The identification of a petroleum product is made by comparison of the standard analyzed under the same conditions versus sample chromatogram on the FID, utilizing pattern recognition techniques.

NOTE: The low level procedure for soils is SW-846 Method 5035. Some agencies and programs are requiring the use of SW-846 Method 5030 for soil analyses. The procedures for each method are included in this SOP. The default for low level soils will be 5035.

2.2 This procedure is based on the guidance in SW-846 Method 8015B. It is not the intention of this SOP to replace the procedures mandated by several state agencies (Tennessee, Florida, California, Wisconsin, etc.,) although this procedure will produce comparable results.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The analyst should wear a lab coat or apron, eye protection(lab glasses or face shield) and gloves when preparing standards and handling samples. Samples and standards should be handled under a hood or in a well ventilated area.

3.3 The analyst should be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in the lab. The MSDS list the potential hazards that each material poses and information about safe handling of the material.

4.0 INTERFERNCES

4.1 The patterns produced by samples that have been subjected to the conditions of the environment may not be the same as the standards. This is generally referred to as "weathering"-the degradation of the product due to evaporation and/or biological or chemical reactions. A sample with more than one product may also be difficult to identify due to overlap of the peaks.

4.2 Contamination may occur from diffusion of VOCs into the sample during shipping and storage. Fluorocarbons and methylene chloride may penetrate the Teflon septa of the sample vial. Sampling and storage areas should be as free from volatile contamination as possible. Trip blanks should be prepared and analyzed to check for this type of contamination.

4.3 Care must be taken to ensure that the analysis area is also free of aerosols and vapors. Contaminants in the air can diffuse into samples during preparation and loading. Many spray cleaners, typewriter cleaners, paints, varnishes, and furniture and carpet cleaners have solvents that may affect the VOC analysis.

4.4 Contamination by carryover can occur when high level and low level samples are run sequentially. Cross contamination can also occur from syringes and purge tubes that are not properly cleaned and rinsed. Purge tubes should be cleaned with hot, soapy water, rinsed with tap water, and then dried at 105°C until dry. Syringes should be cleaned by rinsing repeatedly with methanol.

4.5 Some care should be taken in the maintenance of the Teflon ferrules and seals used on the purge and trap concentrators.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

5.1 Liquid samples are collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are acidified at the time of collection with about 0.10mL of concentrated HCl per 40mL of sample. The acid prevents the biological degradation of the aromatic compounds and prevents the dehydrohalogenation of some of the chlorinated alkanes. The sample must be iced or refrigerated at 4C+/- 2C from the time of collection until analysis. The holding time for samples preserved with HCl is 14 days for all target compounds. The holding time for unpreserved samples is 7 days.

5.2 Soils: Soils are routinely collected in duplicate in Encore samplers. A "bulk" sample is also routinely collected in a 125-mL jar fitted with Teflon-lined caps. The bulk sample can be used for direct purging by SW-846 Method 5030 or may be used to prepare the methanol extraction if the concentration exceeds the working range of the analytical system.

Soils collected in Encore samplers must be analyzed within 48 hours of collection or must be transferred within 48 hours to a sealed vial containing sodium bisulfate solution to preserve the sample. The hold time of the preserved sample is 14 days from the date of collection. The procedure for preparing soil samples is given in Section 9.2.

5.3 High level soil and waste samples are collected in glass containers (usually 125-mL clear glass) equipped with Teflon-lined caps. Soil samples may also be submitted as core samples contained in metal or plastic "tubes" or in 40-mL VOA vials. The samples are iced at the time of collection and stored at 4C+/-2C. The holding time for soil and waste samples is 14 days from date of collection.

NOTE: Samples that are suspected of having very high concentrations of VOC should be segregated from the "routine" samples and stored in a manner that will minimize sample and laboratory contamination. See SL-SOP AN70. If possible, keep the field QC in the same storage refrigerator as the samples.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC)-temperature programmable equipped with a flame ionization detector (FID) or flame ionization detector and photoionization detector (PID).

6.2 Purge and trap(P/T) system compatible with the GC, consisting of a concentrator and compatible autosampler.

6.3 Data system or integrator compatible with the GC and P/T device

6.4 Supelco "J" trap for BTEX. This trap is recommended because it does not retain as much methanol as the traps containing charcoal or silica gel. This is a definite advantage when the FID is used for identification and quantification of targets

6.5 Recommended Columns and Column configuration-
DB-VRX fused silica capillary column, 60 m X 0.45 mm i.d., 2.5 mm film
DB-624 fused silica capillary column, 60 m X 0.53 mm i.d., 3.0 mm film
DB-Stabilwax fused silica capillary column, 60 m X 0.53 mm i.d., 3.0 mm film

The recommended configuration is connecting two dissimilar columns to the transfer line of the P/T or injector port with a glass y-splitter. One column is connected to the FID and the other column to the PID. The Stabiliwax should be connected to the PID if it is used. This column will provide separation between the xylene isomers and styrene. This configuration provides simultaneous detection and confirmation of the target compounds.

The GC may also be configured with a single column connected an FID or to a PID in series with an FID.

6.6 Gastight microsyringes and syringes, appropriate volumes.

6.7 Solution storage container, glass with Teflon-lined screw caps. The volume of the container should allow storage of the container with a minimum of headspace.

6.8 Top-loading balance

7.0 REAGENTS

Reagents must be tracked in accordance with SL SOP AN44:*Reagent Traceability*.

7.1 Methanol: Burdick and Jackson Purge and Trap Grade Methanol

7.2 Reagent water-lab generated deionized water or deep well water that has been purged with nitrogen. The purging vessel may be heated to facilitate the removal of VOC from the water.

7.3 Sodium bisulfate-reagent grade. This salt is hydroscopic and should be stored in a dessicator.

7.4 Soil preservation solution- Slowly add, while stirring, 200g of sodium bisulfate to a 1.0-L volumetric containing about 700mL of reagent water. After the salt has dissolved, dilute to volume with reagent water, transfer to a storage container, and store the solution in an area free from VOC-especially water soluble solvents such as acetone.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all stock solutions or neat compounds. All preparation steps must be in accordance with SL SOP AN41: *Standard Material Traceability*.

8.1 Preparation of Calibration Standards

8.1.1 Preparation of Stock Standards from Neat Compounds

The lab should attempt to obtain a certified primary standard or secondary standard before preparing stock standards from neat materials. SL-SOP AN43: *Standard Preparation* gives the general instructions for the preparation of the stock solutions from neat materials.

Neat petroleum products should be used when provided as site specific materials. When preparing standards of a general nature (i.e. Gasoline), these materials should be purchased from a reputable standard vendor. Gasoline is available as a certified stock standard from Restek and should be used unless a site -specific material is provided. Stock standards of site-specific products should be prepared from neat materials at a concentration of 2000-5000ug/mL in methanol.

8.1.2 Preparation of the Working Standard from Stock Standards

The working standard is prepared from the primary stock standards that are either prepared from neat compounds or purchased as certified solutions. The working standard contains one or more of the target compounds at a concentration suitable for preparing the calibration standards, generally 100-200ug/mL. A known volume of the working standard is then added to a known volume of reagent water to make the calibration standard.

The volume of stock standard needed to prepare a working standard is given by the following equation:

$$Cs \otimes Vs = Cws \otimes Vws$$

$$Vs = \frac{Cws \otimes Vws}{Cs}$$

where

Cs = concentration of stock standard(ug/mL)
Vs = volume of stock standard needed (mL)
Cws = concentration of the working standard(ug/mL)
Vws = volume of working standard to be prepared (mL)

EXAMPLE
Prepare 1.0mL of a 25ug/mL working standard from a 2000ug/mL stock standard.

$$Vs = \frac{Cws \otimes Vws}{Cs} = \frac{25ug/mL \otimes 1.0mL}{2000ug/mL} = 0.0125mL = 12.5uL$$

12.5uL of the 2000ug/mL stock standard is diluted to 1.0mL with methanol. The concentration of the working standard is 25ug/mL.

The standards and standard concentrations listed in Appendix A are recommended for routine use. If other "recipes" are used, the lab must document the standard preparation procedures in the standard traceability log.

8.1.3 Calibration Standards

A five point calibration curve is prepared and analyzed for gasoline and any site-specific product. The lowest concentration calibration standard must be at or below the reporting limit and the rest of the standards will define the working range of the detector.

If multiple products are being analyzed, the curves for all targets are verified by the analysis of the gasoline mid-level standard. For example, if gasoline and lacolene are being determined, a five point curves of both products are analyzed initially but only gasoline is analyzed to verify the curve for both products; that is, if gasoline meets the CCV criteria, lacolene is assumed to meet the criteria.

An example of the preparation of calibration standards are given in Appendix A. IF the lab uses a different "recipe" to prepare the calibration standards, the recipe must be documneted in either the traceability log or as a controlled posting.

The lab may use either or both of two compounds for surrogates: a,a,a-trifluorotoluene or 2,5-dibromotoluene. The advantiage to using 2,5-dibromotoluene is that it elutes after naphthalene and will be subjet to less matrix interfernece than a,a,a-TFT. 4-BFB is used as the ISTD.
The surrogates may be calibrated as part of the gasoline calibration (example in Appendix A) or may be calibrated separately.

8.2 Matrix Spiking Solutions

Gasoline is utilized as the matrix spiking compound. Prepare a solution at a concentration of 1000ug/mL. The addition of 10uL of this solution will result in the following concentrations(Ct):

Aqueous:

$$Ct = \frac{500ug/mL \otimes 0.010mL}{0.025L} = 200ug/L$$

Soils:

$$Ct = \frac{500ug/mL \otimes 0.010mL}{0.005kg \otimes solids} = \frac{1000ug/kg;dw}{solids}$$

solids = (percent solids)/100

9.0 SAMPLE PREPARATION

Composite samples can be prepared using the guidance provided in SL-SOP AN71: *Compositing of Samples.*

9.1 **Aqueous samples** are analyzed directly by purge and trap/GC-MS. No sample preparation is necessary except to homogenize the sample prior to subsampling. The pH of liquid samples is checked and recorded prior to analysis to determine if the sample has been properly preserved.

9.2 Preparation of Soil Samples (5035)

9.2.1 Remove the Encore samples and the bulk sample from the storage area.

9.2.2 Test an aliquot of the bulk sample for the presence of carbonates.

- Transfer 5g of sample from the bulk sample to a 40mL vial..
- Add 5ml of the sodium bisulfate solution and shake the vial .
- If the sample exhibits effervescence, the Encore samples should be preserved as described above using 5mL of volatile-free water in place of the sodium bisulfate solution and placed in a freezer at –10C. The analytical hold time for frozen samples is 14 days from collection.
- If no effervescence is noted, the Encore samples may be preserved with 5mL soil preservation solution.

9.2.3 Add a stir bar to a vial and weigh the vial and record its tare weight(or tare the vial and stir bar weight by pressing the autotare button).

9.2.4 Transfer the sample from the Encore sampler to the tared vial and record the weight of the sample log.

If the sample effervesced during the carbonate test (9.2.2), add 5.0mL of reagent water and freeze at –10C. The hold time is 14 days from collection

If not, add 5.0mL of the soil preservation solution, seal the vial, and store the sample at 4C until the time of analysis. The preserved sample must be analyzed within 14 days of collection.

NOTE: A preparation blank is prepared when Encore samples are transferred. The preparation blank contains the same reagents as the samples-either 5mL of reagent water or 5mL of soil preservation solution.

9.2 High Level Soils

Soil samples which contain VOC at levels which exceed the working range of the calibration (5035) or require the analysis of less than 1.0g (5030) must be analyzed using the methanol extraction procedure. Volatile screen data will indicate whether a low or high level analysis is required. If screen data is not available, historical information or the characteristics of the sample may be used as an aid in determining the proper analysis level. It is better to err on the conservative side and analyze the sample by the high level method first to avoid the possible contamination of the entire analytical system.

9.3.1 Remove the sample from the refrigerator and allow the sample to equilibrate to ambient temperature.

9.3.2 Carefully remove the cap from the sample container. Using a clean glass rod or stainless steel spatula, mix the sample thoroughly to produce a homogeneous sample. If water is present on top of the sample, check with the project manager to determine if the water should be decanted off. Normally, the water is mixed into the sample.

9.3.3 Weigh out 10g +/-0.1g of the homogenized sample into a 12-mL VOA vial using the top-loading balance. Avoid getting rocks or leaves into the sub-sample. Record the weight of the sample in the VOC extraction log or instrument log to the nearest 0.1g. Weight two additional 10-g aliquots of the sample selected as the MS/MSD.

9.3.4 Wastes: Weigh out 1g +/-0.1g of the homogenized sample into a 12-mL VOA vial using the top-loading balance. Record the weight of the sample in the VOC extraction log or instrument log to the nearest 0.1g.

9.3.5 Quickly add 10mL of methanol. Add 30uL of the 2000ug/mL surrogate stock solution to each sample. Cap the vial and shake the vial for 30 seconds

The theoretical concentration of the surrogate in 10g of sample is:

$$Ct = \frac{2000ug/mL \otimes 0.030mL}{0.010kg} = 6000ug/kg$$

9.3.6 The method blank is prepared by adding 10mL of methanol to a vial or to vial containing 10g of blank sand. Add 30uL of the surrogate stock solution (2000ug/mL) to the vial and shake the vial for 30 seconds.

9.3.7 The LCS is prepared by adding 10mL of methanol to a vial or to 10g of blank sand in a vial. Add 30ul of the surrogate stock solution (2000ug/mL) and 200uL of the gasoline stock solution (2500ug/mL) to the vial and shake the vial for 30 seconds. The MS/MSD is prepared in the same manner on the duplicate samples.

The theoretical concentration of the LCS/MS in 10g of sample is:

$$Ct = \frac{2500ug/mL \otimes 0.200mL}{0.010kg} = 50000ug/kg$$

9.3.8 After the soil/solid portion of the sample has settled out, transfer a portion of the extract to a vial. The extract must be stored with no headspace at 4C until the time of analysis. Discard the remaining extract.

9.3.9 A portion of the methanol layer is injected into a 25mL of reagent water. The volume of the aliquot should not exceed 125uL. 5uL of the internal standard mix (150ug/mL) is added to the sample and purged in the same manner as liquid samples (ambient purge conditions).

10.0 PROCEDURE

10.1 Instrument Conditions

10.1.1 GC Conditions

GC conditions may vary according to the environment and condition of each instrument. The lab must document the instrument conditions to assure consistent results and to aid in trouble-shooting the analytical system. Each lab is responsible for assuring that the conditions necessary to achieve adequate separation and sensitivity of the target analytes are maintained.

10.1.1.1 Example GC temperature program(ambient cooling)

Initial column temperature: 35 C for 3 minutes
Column temperature program 1: 20C per minute
Intermediate column temperature: 70C for 4 minutes
Column temperature program 2: 10C per minute
Final column temperature: 200C for 5.25 minutes

10.1.1.2 Column flow: Approximately 5-10mL/minute helium with a make-up of 20-25mL/minute helium.

10.1.2 Purge and Trap Conditions

The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes and the desorb time should be at least 2 minutes. Other parameters may be varied to optimize the detection of the target compounds.

10.1.2.1 "Three ring trap"-charcoal, Tenax, silica gel
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 180C
Bake time: 8 minutes at 225C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.1.2.2 BTEX Trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 225C
Bake time: 8 minutes at 250C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.1.2.3 VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 225C
Bake time: 8 minutes at 250C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.2 Calibration
The analytical system is calibrated initially by the analysis of a five point curve for gasoline or the site-specific product. A GRO standard is analyzed daily to set the Retention time windows for the GRO constituents. The GRO standard may also be used to check the calibration of the BTEX compounds if these compounds are being measured concurrently with the petroleum products. If the BTEX compounds are also to be determined using the same analysis, the guidelines in SL SOP VG01 (602 or 8020) or SL SOP VG02 (8021B) must be followed.

Internal or external standard calibration techniques may be employed for the determination of the concentration of GRO and volatile petroleum products. The internal standard technique is recommended and included in this SOP.

10.2.1 The initial calibration for identification and quantitation of products will involve the analysis of a five point calibration for either gasoline or the site-specific product. and single point calibration standards for the carbon range standard.

10.2.1.1 Prepare the calibration standards using the guidelines listed in Section 8 of this SOP and analyze the standards using the instrument conditions that are optimized for the analysis. Any required maintenance should be performed prior to analysis of the initial calibration.

10.2.1.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?

>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?

>Are the peaks symmetrical? Is there tailing or fronting?

>Are the areas of the peaks normal for the sensitivity setting being used?

10.2.1.3 Tabulate the total areas for each of the five initial calibration standards (either the gasoline or the site-specific fuel). Quantitation of sample concentration for the multi-component products is based on the total area (including baseline integration) of all the peaks in the retention time range defined by the reference petroleum standard.

10.2.1.4 A calibration curve can be established for each analyte by plotting the concentration (or nanograms on-column) along the x-axis and the corresponding response along the y-axis. A regression curve is prepared and evaluated. A curve with a 0.99 or higher correlation coefficient can be used for quantitation.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPs for other exceptions to using non-linear curve fitting.

10.2.1.5 Alternatively, calculate the relative response factor (RRF) for each calibration standard using the following equation:

$$RRF = \frac{Ac}{Aist} \otimes \frac{Cist}{Cc}$$

where
Ac = total area of the compound or petroleum product
Aist = area of the internal standard
Cist = concentration of the internal standard (ug/L or ug/kg)
Cc = concentration of the target compound or petroleum product (ug/L or ug/kg)

10.2.1.6 Calculate the average response (or calibration) factor for the five point diesel or site specific product.

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 + RF_4 + RF_5}{5}$$

10.2.1.7 Calculate the standard deviation of the five calibration levels for each target.

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}}$$

where
RF_i = response factor of the individual calibration level
RRFavg = average response factor

10.2.1.8 Calculate the relative standard deviation (% RSD) of the five levels for each target:

$$\%RSD = \frac{standard\ deviation}{\overline{RF}} \otimes 100$$

If the % RSD is less than 20% for the gasoline or site-specific product, the calibration is considered linear and the average response factor (or calibration factor) is used for quantitation.

10.2.2 The continuing calibration check standard will be either the mid-level gasoline standard (if multiple products are being determined) or the mid level site-specific product standard if this single product is being determined. Choose one of the following to evaluate the calibration check.

10.2.2.1 Analyze the mid level gasoline or site-specific product. Tabulate the total area of the target analyte and calculate the response factor in the same manner as the initial calibration (10.2.1.4).

Calculate percent difference(%D) of between the initial and continuing calibration response factors:

$$\%D = \frac{RF_{init} - RF_{cont}}{RF_{init}} \times 100$$

where

RF_{init} = average response factor from the initial calibration curve

Rf_{cont}= response factor from the continuing calibration standard

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If the percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor is used for quantitation.

If the percent difference is greater than 15%, another continuing calibration standard should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the continuing calibration criteria is not met, a new initial calibration must be prepared and analyzed.

10.2.2.2 If the calibration curve option has been chosen, calculate the percent drift (%D) of between the initial and continuing calibration response based on the true or theoretical concentration of the check standard:

$$\%D = \frac{result - expected}{expected} \times 100$$

where

result = concentration or nanograms on-column of the calibration check standard quanted against the curve

expected = true concentration or nanograms on-column of the calibration check standard

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If the percent drift is less than or equal to 15%, the calibration curve is verified and can be used for quantitation.

If the percent difference is greater than 15%, another continuing calibration standard should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the continuing calibration criteria is not met, a new initial calibration must be prepared and analyzed.

10.3 Analytical Sequence

Initial Calibration Standards -5 point gasoline or site specific product -1 point carbon range standard (GRO)
Method Blank
Sample analyses (up to 12 hours)
Continuing calibration verification-mid point gasoline standard or site-specific product* – Performed @ 12 hours from last CCV
Method Blank
Sample analyses (up to 12 hours)
Continuing calibration verification-mid point gasoline standard or site-specific product* – Performed @ 12 hours from last CCV
Method Blank
Sample analyses (up to 12 hours)

The sequence continues until all samples are analyzed or until the calibration verification standard fails the acceptance criteria.

*If multiple products are being determined, gasoline may be used to verify the calibration for all products; if gasoline passes, all of the others are assumed to pass. If only one product is being determined, that product is used to verify the calibration.

10.3.2 Aqueous Sample Analysis

Take the samples out of the refrigerator and allow them to equilibrate to room temperature.

Invert the sample vial several times to insure a homogenous mixture.

Remove the cap from the sample vial and gently pour the sample into a 25-mL syringe.

Spike the sample with 25 uL of the F_3Toluene surrogate working standard and then load into a sparge tube at the purge and trap autosampler through the sample sparge needle/valve assembly.
All samples requiring a spike must also have 10 uL of the gasoline or petroleum product working standard added to them. This will result in a spike concentration of 200 mg/L.

Adjust the valve to insure that it is in the proper position for purging. Any aqueous dilutions are performed by adding the appropriate aliquot of the sample to reagent water in a 25 mL syringe to yield a final volume of 25 mL.

Analyze the sample under the conditions optimized for ambient purge of liquids. (See Section 10.1 for the recommended instrument conditions).

10.4. Low Level Soil Samples by Heated Purge and Trap (Method 5035)

10.4.1 Remove the samples to be analyzed from the refrigerator and allow the sample to come to ambient temperature. Inspect the vial for cracks or obvious breaches in the septum.

10.4.2 Load the samples on to the soil purging unit. Standards and QC items must be analyzed under the same heated purge and trap conditions.

10.5 Low Level Soil Samples by Heated Purge and Trap (Method 5030)

The sealed, tared vial containing the sample is removed from the refrigerator and allowed to equilibrate to room temperature. The weight of the sample is determined (Section 9.3) and the sample is loaded on to the soil autosampler. Standards are prepared and analyzed under the same conditions. Note that the soil autosampler will automatically add reagent water and the internal standards to each vial.

If a smaller aliquot of sample is required because of the high concentrations of target compounds, refer to section 9.3.

10.5.1 Remove the samples to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.5.2 Put on a pair of gloves before transferring the sample from the vial to the sparging vessel. The sample may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. ***Gloves must be worn when handling samples.***

10.5.3 Mix the contents of the container by stirring the sample with a spatula or glass rod. The sample must be homogenized before an aliquot is weighed into the sparging vessel. Weigh 1g to 5g of the sample into the sparging vessel. Attach the sparging vessel to the autosampler. Record the weight of sample, to the nearest 0.1g, on the analysis log. Do not use less than 1g of sample for the low level soils.

10.5.4 Remove the plunger from the glass syringe. Attach a syringe valve to the syringe Luer-tip to prevent reagent water from spilling out of the syringe. Add reagent water to the syringe and adjust the volume to 5.0mL.

10.5.5 Open the syringe valve and inject the internal standard/surrogate (ISSU) mix into the reagent water. Add the reagent water with the ISSU mix to the sparging vessel.

10.5.6 Attach the heater jacket or other heating device to the sparging vessel and analyze the samples using the same conditions used for the initial and continuing calibration standards-purge temperature for low level soils is 40C. Record the sample information on the analysis log.

10.5.7 Determine the concentration of the samples and QC items. If the concentration of a target compound is above the highest calibration standard, a smaller sample aliquot is weighed and analyzed. Do not use less than 1g of sample for the low level soils. If the concentration of a target compound exceeds the linear range using a 1-g aliquot of soil, the medium level soil extraction must be used.

10.6 Analysis of Methanol Extracts of Soils/Solids

The methanol extraction is used when the concentration of one or more target compounds exceeds the linear range of the low level purge technique. Samples are analyzed only after the initial or continuing calibration criteria has been met, and the method blank criteria has been met. Medium level soil extracts are quanted using the ambient purge calibration curve. Sample preparation steps are included in Section 9.

Remove the plunger from the 25-mL syringe and fill the barrel to overflowing with reagent water(syringe valve in the "red" position).

Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume (5mL)

Briefly remove the syringe valve and inject the sample extract (maximum of 125uL) and 25uL of the internal standard (IST) solution into the syringe.

NOTE: Use the internal standard (IST) mix when preparing the medium level samples. Recall that the surrogates have already been added to the sample prior to methanol extraction.

Load the sample on to the purge and trap device and begin the analysis. All pertinent information concerning the samples must be recorded on the analysis log. The samples must be clearly identified and traceable to the extraction log. These conditions must be the same as was used for the initial and continuing calibration standards-ambient purge for aqueous samples.

Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, a smaller aliquot of the methanol extract is reanalyzed.

NOTE: It is possible to dilute the surrogates in the sample extract below the linear range of the calibration curve. The minimum extract aliquot that can be used to provide a quantifiable result for the surrogates and matrix spikes is 0.020mL (20uL). It is not probable that the recovery of the surrogates would be 100%, which is necessary to provide a quantifiable result using a 0.010mL(10uL) aliquot of the extract.

11.0 DATA ANLAYSIS AND CALCULATIONS

11.1 Qualitative analysis

11.1.1 Identification and quantitation of sample concentration is based on the total area (including baseline integration) of all the peaks in the retention time range defined by the reference petroleum standard. The ability to make a positive identification of a specific petroleum product may be inhibited by sample weathering, multi-product composition, and dissimilarity between the sample and the products utilized as standards. Weathered samples will tend to display peaks not found in the reference standard due to compound degradation or may not contain some of the peaks found in the reference standard. Highly weathered samples may also display an elevated baseline resembling a very broad peak or "humpogram." The total area contained in this peak should be used in the quantitative determination if the peak or hump resides within the retention time range of the reference petroleum product.

11.1.2 If an identification is not possible, additional information may be provided by the analysis of the straight chain alkane standard. Based on the retention time of the various alkanes, an estimated hydrocarbon chain length for the major constituents in the sample can be identified. The sample chromatogram may be quantified against the petroleum standard that most resembles the sample product and reported with a qualifier such as :

"The peaks in Sample X elute between C6 and C10 and the sample was quantified against the gasoline standard, which contains peaks in approximately the same carbon range."

11.1.3 The sample must be diluted to point where the peaks are within the linear range of the gasoline or site-specific product or to a point where the total area is within a factor of five of the total area of the target products. Diluting the extract into the proper range will aid in the identification and evaluation of the chromatogram.

11.1.4 If GRO is requested and a state-specific procedure is not specified, the peaks eluting from 2-methylpentane(C6) and 1,2,4-trimethylbenzene(C9) are summed and quantified against the average response factor or curve of the gasoline calibration.

11.1.5 If "total hydrocarbons" is requested, GRO is calculated and reported.

11.2 Calculations for Samples-External Standard Technique

11.2.1 Aqueous Samples

If a regression curve is used, the concentration is given:

$$concentration(ug / L) = concentration(curve) \otimes DF$$

where
DF = dilution factor

If the relative response factor is used, the calculation for samples is :

$$concentration(ug/L) = \frac{Ax}{RRFavg} \otimes DF$$

where
Ax = area of the target compound being measured
RRFavg = average response factor of the compound being measured
DF = dilution factor

The reporting limit (RL) for each sample is given:

$$RL(ug / L) = RLqap \otimes DF$$

where
DF = dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

11.2.2 Soils by Heated P/T

If the regression curve is used, the concentration is given:

$$conc(ug / kg, dw) = Ccurve(ug / kg) \otimes \frac{5.0g}{(W)(solids)}$$

where
Ccurve = concentration from curve(ug/kg)
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg, dw) = \frac{Ax}{RRFavg} \otimes \frac{5.0g}{(W)(solids)}$$

where
Ax = area of the target compound being measured
RRFavg = average response factor of the compound being measured
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The SL CQAP assumes W= 5.0g and solids = 1.

11.2.3 Methanol Extraction Soils and Wastes

If the regression curve is used, the concentration is given:

$$conc(ug, kg, dw) = Ccurve(ug / L) \otimes \frac{Vcal}{(W)(solids)}$$

where
Vcal = volume that calibration curve is based on (0.005L or 0.025L)
W = weight of sample added to the reagent water (g)-defined above(11.2.3.1)

If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg, dw) = \frac{Ax}{RRFavg} \otimes \frac{Vcal}{(W)(solids)}$$

where
Ax = area of the target compound being measured
RRFavg = average response factor of the compound being measured
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = (percent solids)/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)
The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the reagent water (g)
solids = (percent solids)/100)

The SL CQAP assumes W= 5.0g and solids = 1.

11.3 The concentration of VOC in the lab control and matrix spike samples is determined as in Section 11.1. The concentration is compared to the theoretical spike concentration and the percent recovery is calculated.

11.3.1 The theoretical or true value for a spiked sample is calculated using the following equation:

$$concentration = \frac{amount\ of\ compound\ added}{volume\ /\ weight\ of\ sample}$$

For liquid samples, this equation expands to:

$$concentration(ug\ /\ L) = \frac{Cspike \otimes Vspike}{Vsample}$$

where
Cspike = concentration of the spiking solution (ug/mL)
Vspike = volume of spiking solution added to the sample (mL)
Vsample = volume of sample spiked (L)

For soil and solid samples, the equation expands to:

$$concentration(ug\ /\ L) = \frac{Cspike \otimes Vspike}{Wsample \otimes solids}$$

where
Cspike = concentration of the spiking solution (ug/mL)
Vspike = volume of spiking solution added to the sample (mL)
Wsample = weight of sample spiked (kg)
solids = (percent solids)/100

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. Note that the method blank for liquid samples and low level soils is clock-specific and that the method blank for medium level soil samples is batch-specific.

SL-SOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. This criteria is summarized in the attached SOP Summary.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration is the preparation and analysis of spiked reagent water. Section 8 of EPA Method 8000B gives the general procedure for the performance of the IDOC.

12.3 Method Detection Limit

The method detection limit is determined in accordance with SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time.

15.0 REFERENCES

Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC. (Final Update III)

Appendix A
Example Working Level Standards

Gasoline Working Standard

Stock STD	Stock STD conc (ug/mL)	Volume of Stock Std (uL)	Final Volume of Working STD(mL)	conc of Working STD (ug/mL)
Gasoline	2500	200	1.0	500
a,a,a-TFT	2000	12.5		25
2,5-dibromotoluene	2000	12.5		25

Gasoline Range Working Standard

Stock STD	Stock STD conc (ug/mL)	Volume of Stock STD (uL)	Final Volume of Working STD (mL)	conc of Working STD (ug/mL)
GRO	500-1500	50	1.0	25-75

Internal Standard/Surrogate(ISSU) Working Standard

Stock STD	Stock STD conc (ug/mL)	Volume of Stock STD (uL)	Final Volume of Working STD (mL)	conc of Working STD (ug/mL)
a,a,a-TFT	2000	75	1.0	150
2,5-dibromotoluene	2000	75		150
4-BFB stock	2000	75		150

Internal Standard (IST) Working Standard

Stock STD	Stock STD conc (ug/mL)	Volume of Stock STD (uL)	Final Volume of Working STD (mL)	conc of Working STD (ug/mL)
4-BFB stock	2000	75	1.0	150

Appendix A
Example Calibration Standards

GASOLINE CALIBRATION STANDARDS-AQUEOUS

CAL STD	WORKING STOCK conc(ug/mL)	ul of WORKING STOCK	uL of IST	VOLUME OF WATER (mL)	CAL STD conc(ug/L)
gas-1	500	2.5	5	25	50
gas-2	500	5.0	5	25	100
gas-3	500	10	5	25	200
gas-4	500	25	5	25	500
gas-5	500	50	5	25	1000

surrogatesare at 2.5,5,10,25, and 50ug/L; ISTD at 30ug/L each level

GASOLINE CALIBRATION STANDARDS-LOW LEVEL SOILS

CAL STD	WORKING STOCK conc(ug/mL)	ul of WORKING STOCK	uL of IST	WEIGHT OF SOIL (g)	CAL STD conc(ug/kg)
gas-1s	500	2.5	5	5.0	250
gas-2s	500	5.0	5	5.0	500
gas-3s	500	10	5	5.0	1000
gas-4s	500	25	5	5.0	2000
gas-5s	500	50	5	5.0	5000

surrogatesare at 10,25,50,125, and 250ug/kg; ISTD at 150ug/kg each level.

GRO STANDARD-AQUEOUS

CAL STD	WORKING STOCK conc(ug/mL)	ul of WORKING STOCK	uL of IST	VOLUME OF WATER (mL)	CAL STD conc(ug/L)
GRO-1	25-75	20	5	25	20-60

GRO STANDARD-LOW LEVEL SOILS

CAL STD	WORKING STOCK conc(ug/mL)	ul of WORKING STOCK	uL of IST	WEIGHT OF SOIL (g)	CAL STD conc(ug/kg)
GRO-1s	25-75	20	5	5.0	100-300

Appendix B –QC Summary
8015-PURGEABLES METHOD SUMMARY
The primary target for this procedure is gasoline, which is defined as all peaks eluting between 2-methylpentane(C6) and 1,2,4-trimethylbenzene(C9). Gasoline will be quantified using the total area of the peaks eluting in the gasoline range. Other products may be identified and quantified if the calibration criteria is met. BTEX can be determined concurrently.

HOLD/STORAGE
Container: Aqueous: 40mL vial with Teflon-lined cap, no headspace Soil: Pre-weighed vial 40mL vial; a separate sample is also collected in a 40mL or 125mL vial for "dilutions", if required.
Preservative: Aqueous: If chlorine is present, add sodium thiosulfate Soils: 5.0ml sodium bisulfate solution; no preservative in separate soil sample.
Storage: 4C +/-2C from collection to analysis
Hold Time: Aqueous: 14 days if preserved at collection with HCl to pH<2; 7 days if unpreserved. Soils: 14 days

ANALYSIS
Aqueous-ambient P/T; GC with PID/FID
Soils-heated (40C) P/T; GC with PID/FID

ANALYTICAL SEQUENCE

Initial Calibration Standards -5 point gasoline or site specific product -1 point carbon range standard (GRO)
Method Blank
Sample analyses (up to 12 hours)
Continuing calibration verification-mid point gasoline standard or site-specific product* – Performed @ 12 hours from last CCV
Method Blank
Sample analyses (up to 12 hours)
Continuing calibration verification-mid point gasoline standard or site-specific product* – Performed @ 12 hours from last CCV
Method Blank
Sample analyses (up to 12 hours)

The sequence continues until all samples are analyzed or until the calibration verification standard fails the acceptance criteria.

*If multiple products are being determined, gasoline may be used to verify the calibration for all products; if gasoline passes, all of the others are assumed to pass. If only one product is being determined, that product is used to verify the calibration.

SURROGATE(S):
a,a,a-Trifluorotoluene- 30ug/L(aqueous); 150ug/kg(soils)

BATCH QC
Method blank
LCS-gasoline at 250ug/L(aqueous); 1250ug/kg(soils)*
MS/MSD- gasoline at 250ug/L(aqueous); 1250ug/kg(soils)*
*(or site specific target at 5-10X RL)

Appendix B QC Summary

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration- 5 point minimum with lowest point at RL	Prior to sample analysis or when CCV fails	1)RSD of each target <= 20%; OR 2)plot regression curve	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification(CCV) -midpoint calibration standard	Every 12 hours and at end of sequence	1) Within method "Q" table criteria; OR 2) Percent difference or drift <= 15%	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Prior to analysis of samples	All targets reported less than RL in Table 5 of the SL CQAP	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Lab control sample (LCS)- subset of targets in SL CQAP	Per batch (If MS/MSD cannot be performed , the LCS must be performed in duplicate)	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix spike(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogates	All samples, method blanks, and QC	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Reporting limit standard (required for FL DEP)	Daily	Detected at reasonable response	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard -Remake and reanalyze standard -Perform system maintenance and recalibrate
Initial demonstration of Capability(IDOC)-the analyst has to perform the IDOC for either of the analogous 600 or 8000 series methods, not both.	Per analyst	Within the 600- or 8000-series method criteria	-Reanalyze QC sample for the targets that failed to meet the criteria
Method detection limit(MDL)	See CA90	See CA90	-Evaluate data. Check calculations -Reanalyze MDL samples

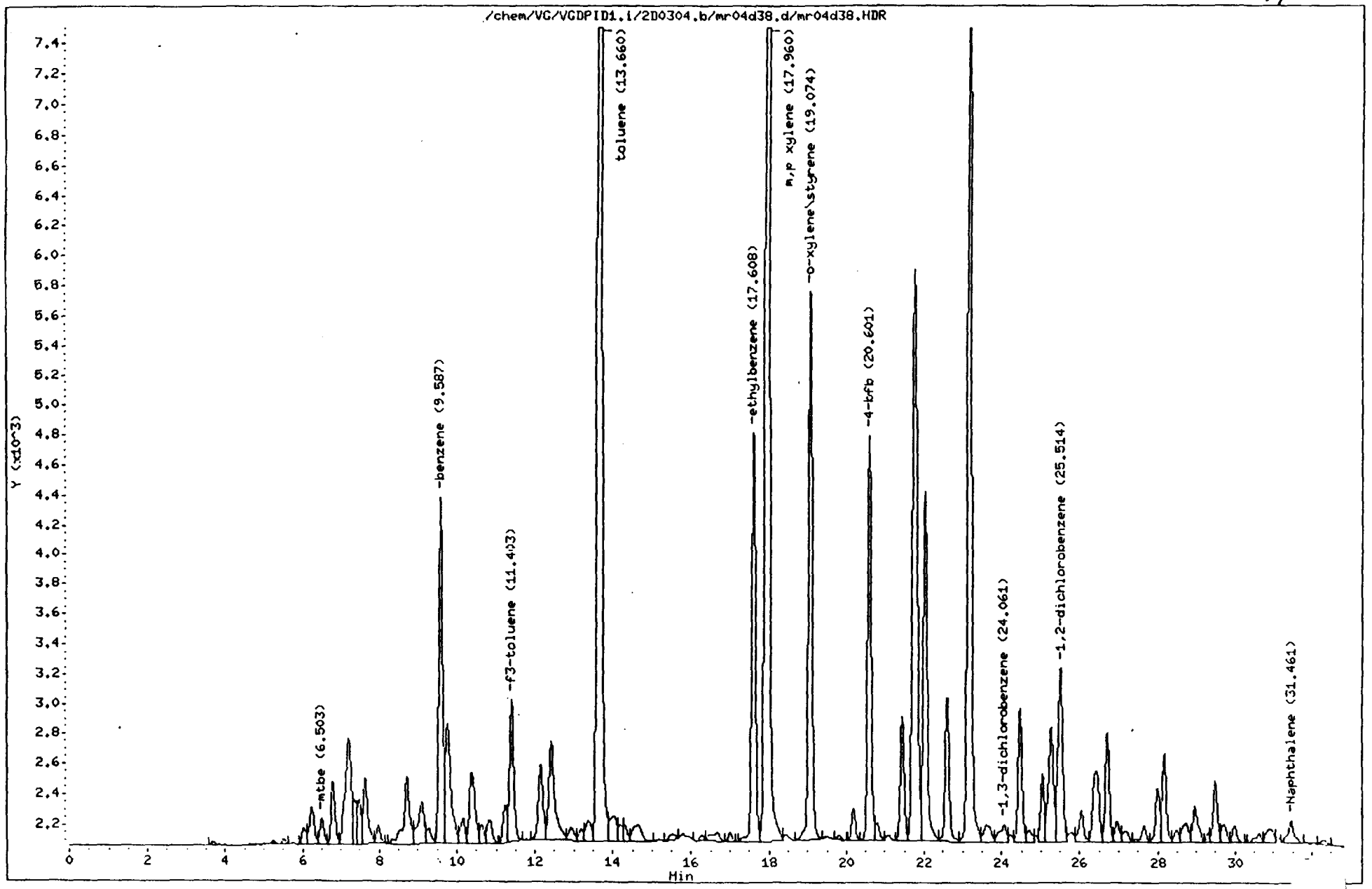
Appendix C
VG15 EXAMPLE CHROMATOGRAMS

CHROMATOGRAM	TARGET	DETECTOR
A	gasoline	PID
B	gasoline	FID
C	mineral spirits	PID
D	mineral spirits	FID
E	lacolene	PID
F	lacolene	FID
G	gasoline + mineral spirits	PID
H	gasoline + mineral spirits	FID

Data File: /chem/VG/VGDPID1.i/2D0304.b/mr04d38.d
Date : 06-MAR-97 08:52:00
Client ID: VGASOL 1.5
Sample Info: VGASOL 1.5~2D0304
Purge Volume: 25.0
Column phase: DB624

Instrument: VGDPID1.i
Operator: VGD
Column diameter: 0.53

A



Data File: /chem/VG/VGDFID2.i/2D0304.b/nr04d38.d

Date : 06-MAR-97 08:52:00

Client ID: VGASOL 1.5

Sample Info: VGASOL 1.5~2D0304

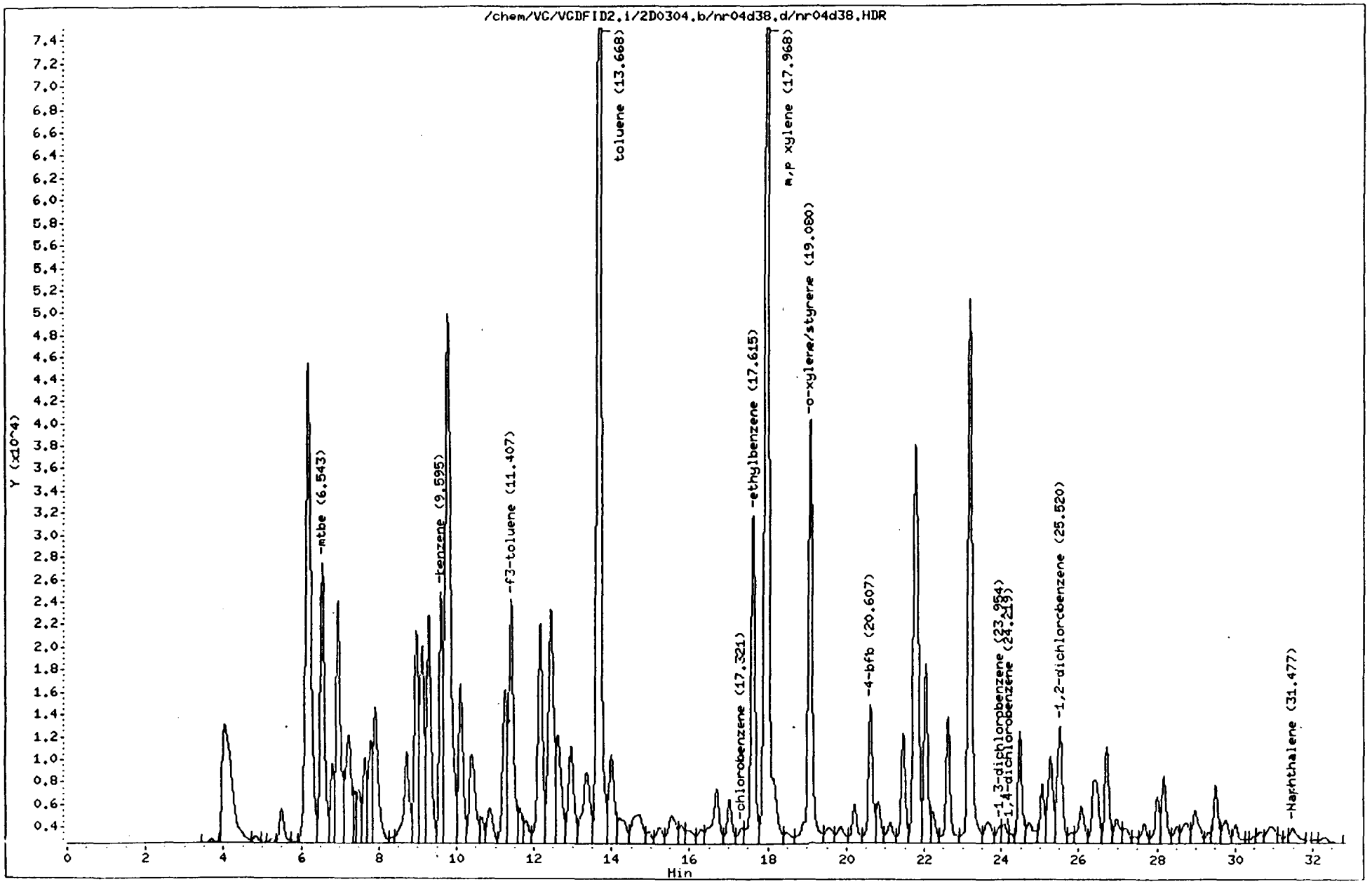
Column phase: DB624

Instrument: VG.i

Operator: VGD

Column diameter: 0.53

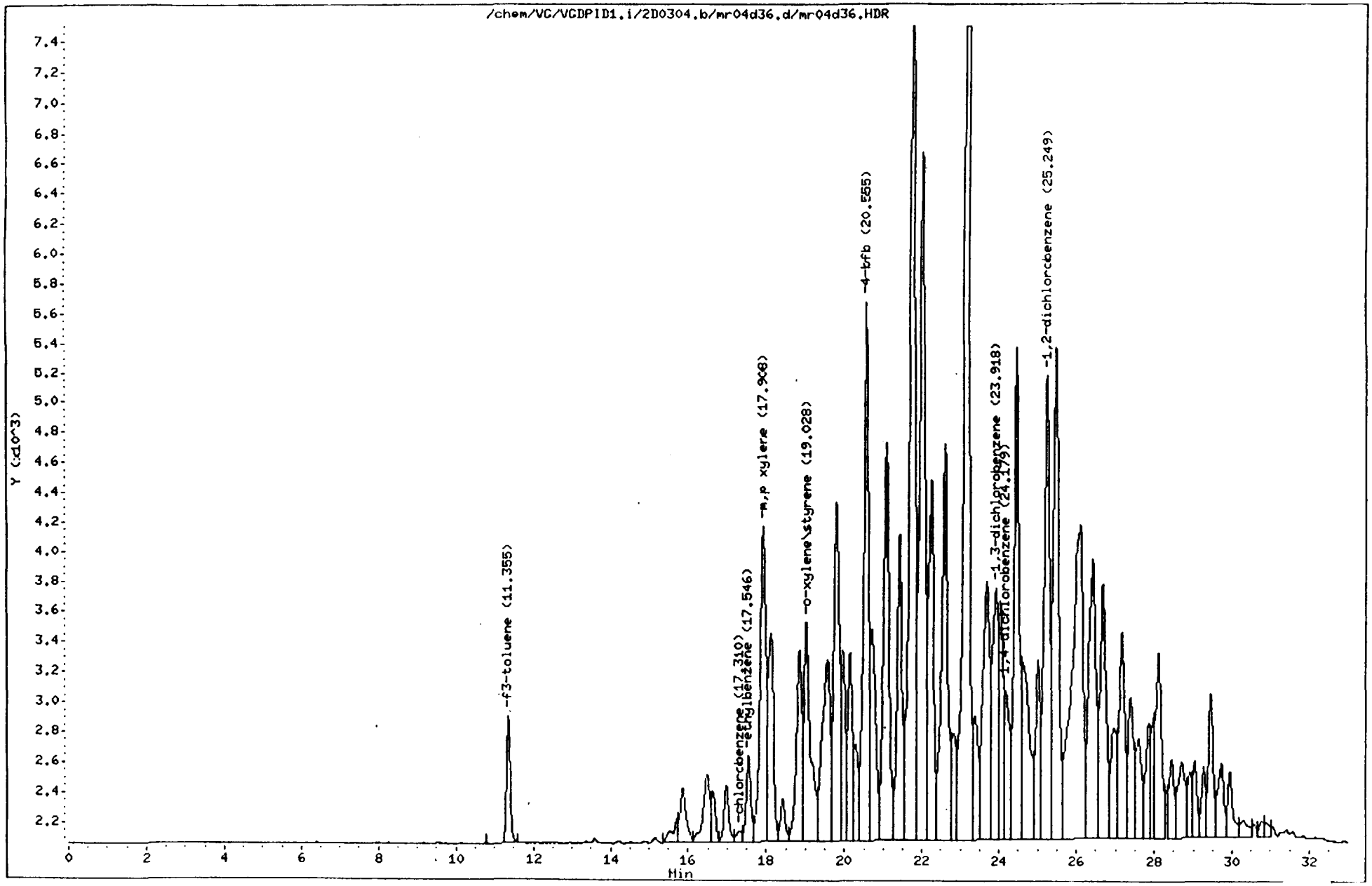
B



Data File: /chem/VG/VGDPID1.i/2D0304.b/mr04d36.d
Date : 06-MAR-97 07:18:00
Client ID:
Sample Info: VHINER-900~2D0304
Purge Volume: 25.0
Column phase: DB624

Instrument: VGDPID1.i
Operator: VGD
Column diameter: 0.53

C



Data File: /chem/VG/VGDFID2.i/2D0304.b/nr04d36.d

Date : 06-MAR-97 07:18:00

Client ID: MINERAL SPIRITS

Sample Info: VMINER-900~2D0304

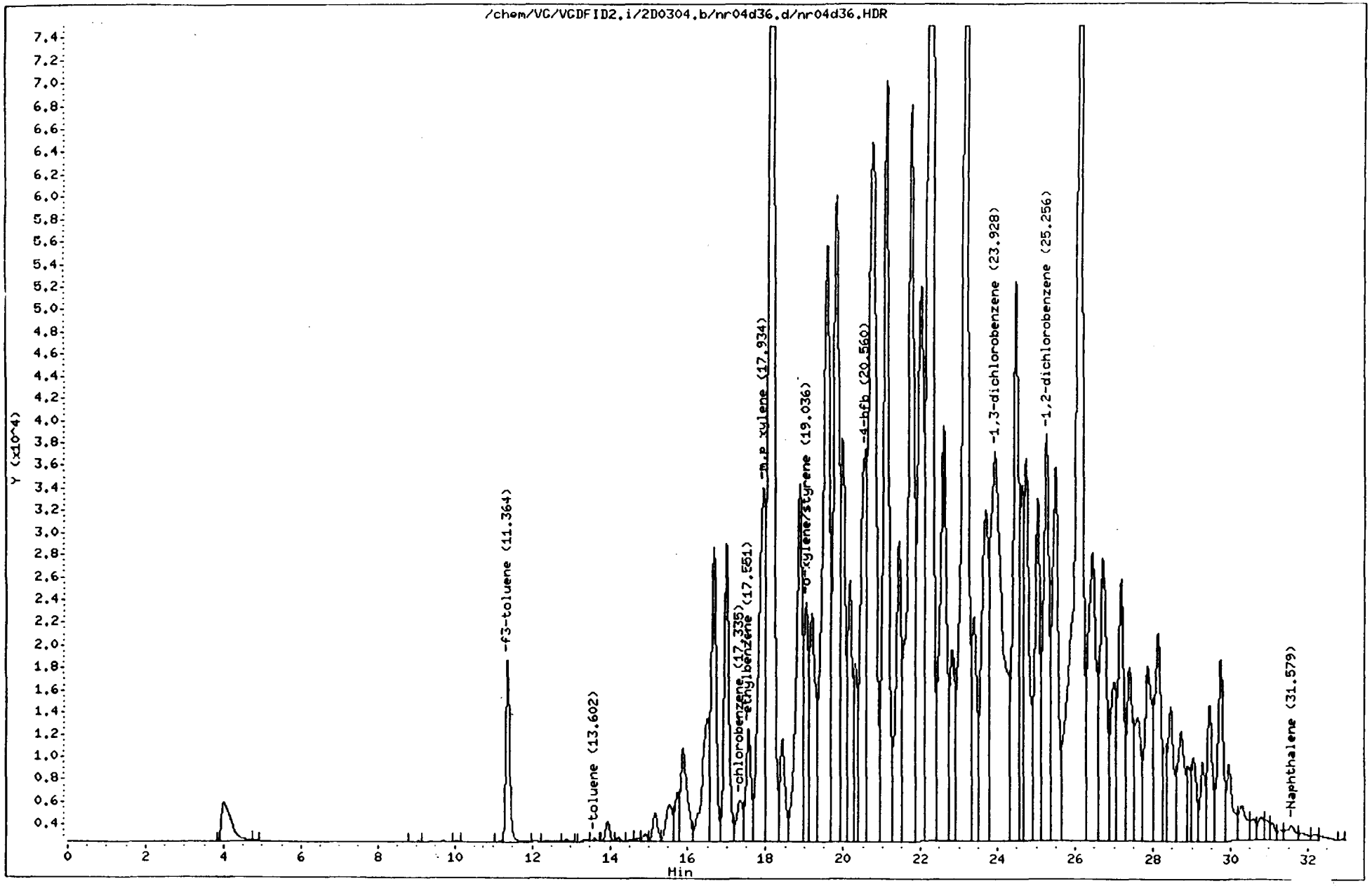
Column phase: DB624

Instrument: VG.i

Operator: VGD

Column diameter: 0.53

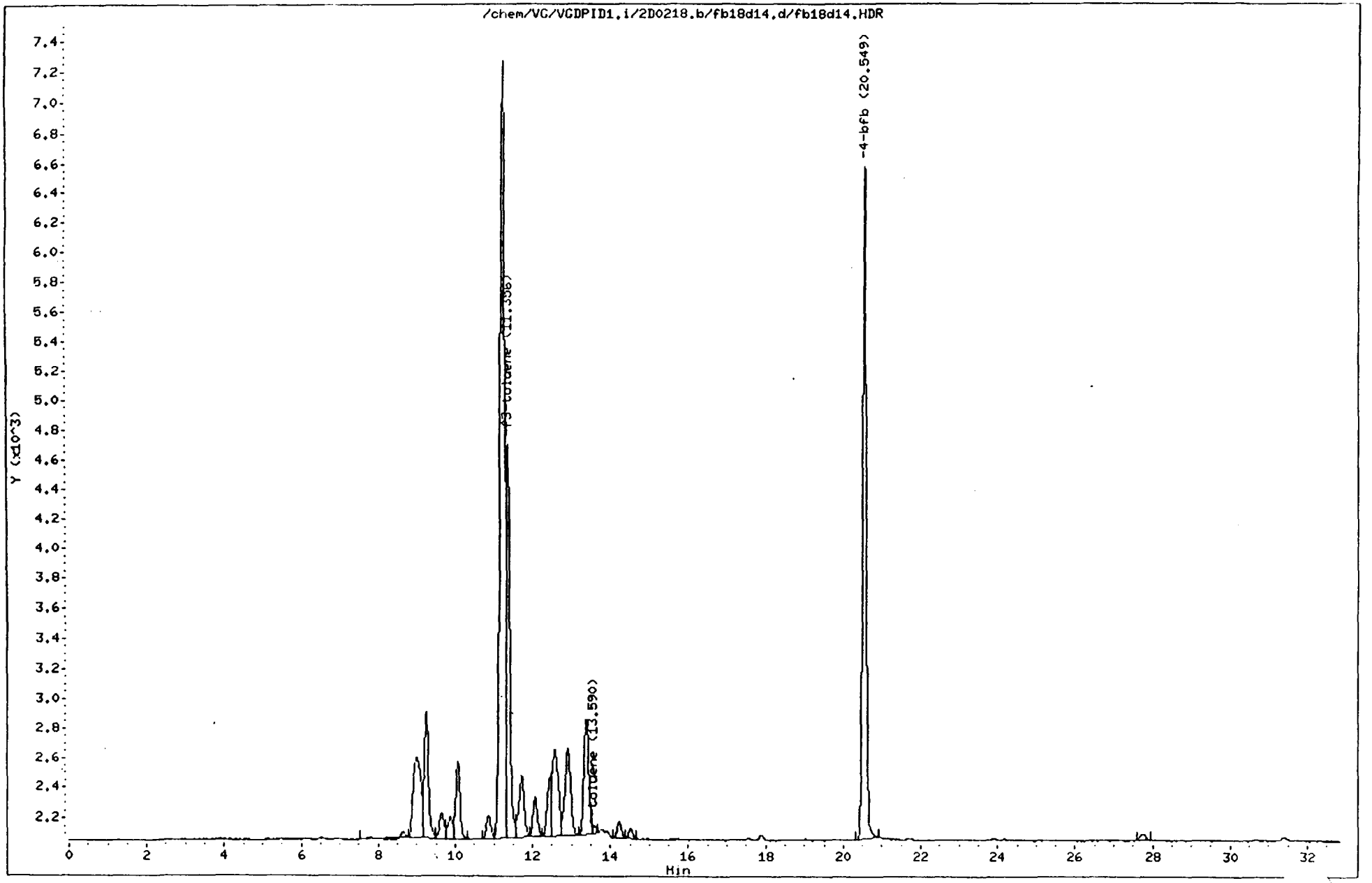
D



Data File: /chem/VG/VGDPID1.i/2D0218.b/fb18d14.d
Date : 18-FEB-97 21:05:00
Client ID: VGLAC-800
Sample Info: VGLAC-800~2D0218
Purge Volume: 25.0
Column phase: DB624

Instrument: VGDPID1.i
Operator: VGD
Column diameter: 0.53

E



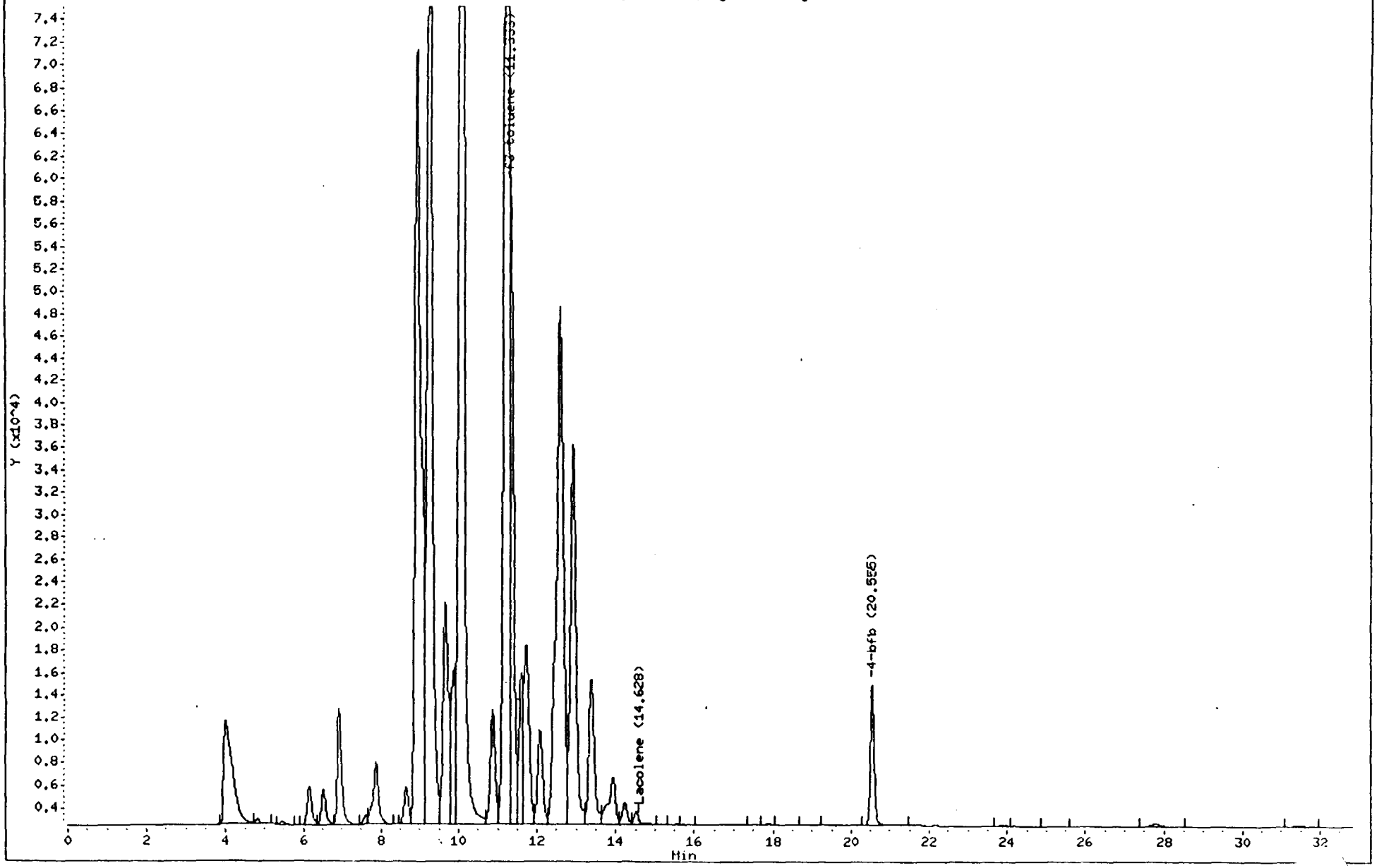
Data File: /chem/VG/VGDFID2.i/2D0218.b/gb18d14.d
Date : 18-FEB-97 21:05:00
Client ID: VGLAC-800
Sample Info: VGLAC-800~2D0218
Purge Volume: 25.0
Column phase: DB624

Instrument: VGDFID2.i

Operator: VGD
Column diameter: 0.53

F

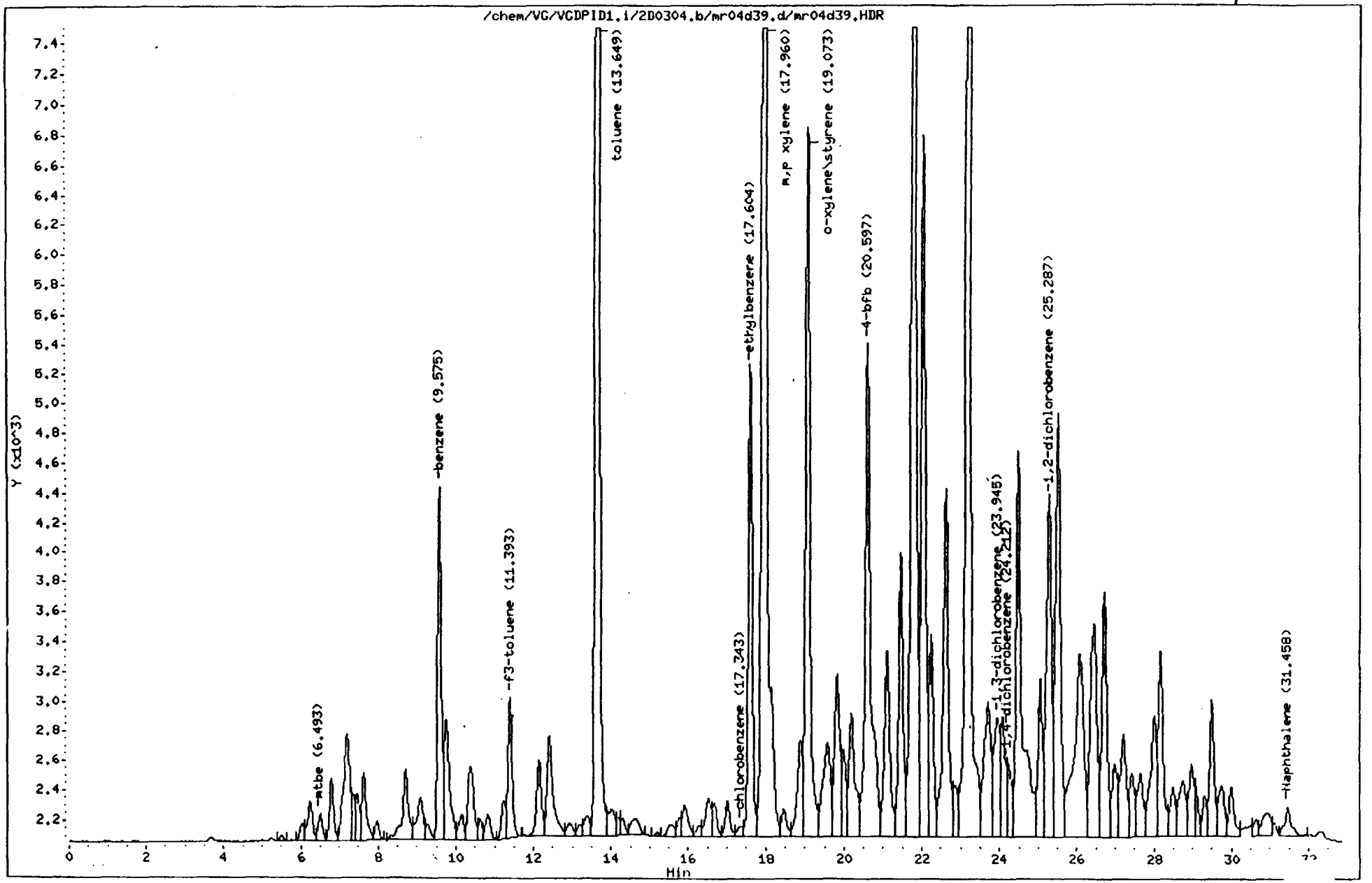
/chem/VG/VGDFID2.i/2D0218.b/gb18d14.d/gb18d14.HDR



Data File: /chem/VG/VGDPID1.i/2D0304.b/mr04d39.d
Date : 06-MAR-97 09:38:00
Client ID: VMI/GS
Sample Info: VMI/GS ~2D0304
Purge Volume: 25.0
Column phase: DB624

Instrument: VGDPID1.i
Operator: VGD
Column diameter: 0.53

G



Data File: /chem/VG/VGDFID2.i/2D0304.b/nr04d39.d

Date : 06-MAR-97 09:38:00

Client ID: VMI/GS

Sample Info: VMI/GS ~2D0304

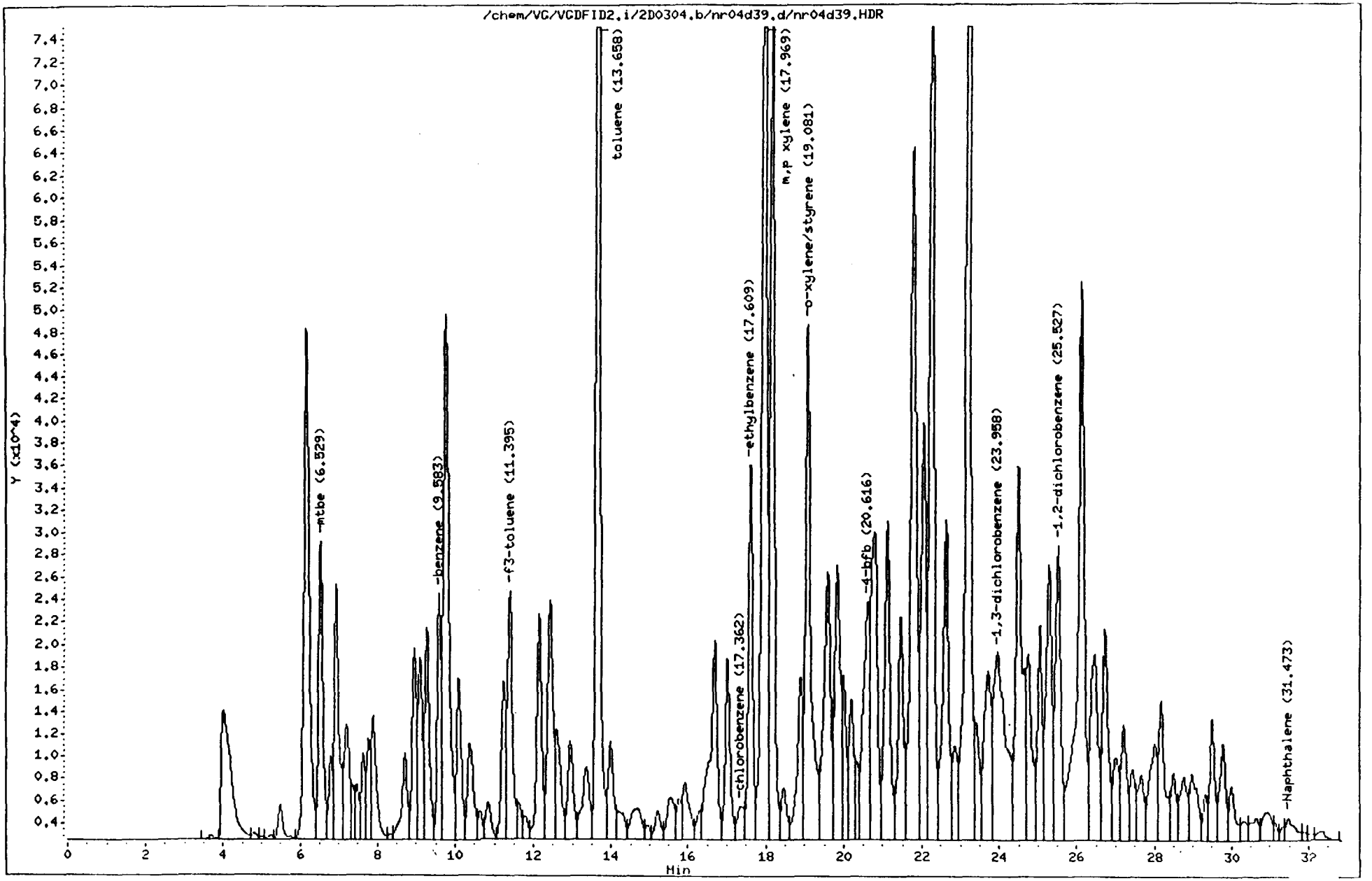
Column phase: DB624

Instrument: VG.i

Operator: VGD

Column diameter: 0.53

H



CHANGE -IN-PROGRESS ATTACHMENT

SOP Document No: VM20:
SOP Description: Volatile Compounds by GC/MS (8260B)

Approval Signature: R. Wayne Robbins	
Title: Corporate QA Manager	Date: Oct 28, 1998

The following revisions or additions have been made to the referenced SOP.

Changes or updates to all sections are in **bold type**.

3.4 **The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of the extract. The traps should be changed a minimum of every three months and disposed of in accordance with SL SOP CA70: *Waste Management.***

6.2 **Gas chromatograph, compatible with the MS and purge and trap systems. If the GC is equipped with an injector that is operated in the split mode, the exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device. The carbon traps should be changed a minimum of every three months.**

Approval Signature: R. Wayne Robbins	
Title: Corporate QA Manager	Date: July 17, 1998

VOLATILE COMPOUNDS BY GC/MS (EPA 8260B)

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of volatile organic compounds (VOC) in water, wastewater, soils/sediments, wastes, oils, sludges, and solids. The attached quantitation report list the target compounds, an example of the retention time order of each target compound, the quantitation and confirmation ions of the target compounds, and internal standard assignments.

1.2 The reporting limit(RL), the method detection limit(MDL), and the accuracy and precision criteria for each target compound are listed in Section 5 of the current revisions of the Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*..

2.0 SUMMARY OF METHOD

2.1 Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the retention time and the relative abundance of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 Aqueous samples may be purged at ambient conditions (recommended) or at 40C (optional). Five to twenty-five milliliter aliquots of the sample may be purged. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

2.3 Low level (<1mg/kg) soil samples are purged at 40C in a purge and trap instrument designed to add water and internal standards to the vial without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards are purged under the same conditions. The minimum weight of sample that can be used for direct purging is 1g.

2.4 High level soils(>1mg/kg) and waste samples are extracted with methanol. A portion of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

2.5 This method is based on the guidance in SW-846 Method 8260B.

2.6 Pentachloroethane is calibrated alone because it breaks down readily to form tetrachloroethene. If pentachloroethane is added to a mixture containing tetrachloroethene, the response of tetrachloroethene is affected. When pentachloroethane is requested, the following calibration procedure will be used:

1) An RL standard will be analyzed in each analytical clock. This standard will be used to determine if pentachloroethane is present in the sample.

2) If pentachloroethane is detected in any sample above the RL, a five point curve is prepared and analyzed and the samples are quantified against the curve. The curve must have a correlation coefficient >=0.99

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedure that you do not understand or that will put yourself or others in a potentially hazardous situation.

3.2 Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, eye protection, or other equipment should be used. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

4.0 INTERFERENCES

4.1 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.

4.2 The volatiles lab must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator blanks should be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in SL SOP AN70:*Segregation of Low and High Concentration Volatile and Semivolatile Samples.*

4.3 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-terachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the lab can clearly demonstrate matrix problems.

4.4 The analysis of highly contaminated samples (>1mg/L or >1mg/kg) can affect succeeding analyses. Carry-over can occur when low concentration samples are analyzed after high concentration samples. Trap replacement and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when carryover is suspected to ensure that the system is free from contamination.

4.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If contamination of the fittings is suspected, the fittings may be heated at 105 C for one hour or replaced.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

5.1 Liquid samples are collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are acidified at the time of collection with about 0.10mL of concentrated HCl per 40mL of sample. The acid prevents the biological degradation of the aromatic compounds and prevents the dehydrohalogenation of some of the chlorinated alkanes. The sample must be iced or refrigerated at 4C+/- 2C from the time of collection until analysis. The holding time for samples preserved with HCl is 14 days for all target compounds. The holding time for unpreserved samples is 7 days.

5.2 Soils: Soils are routinely collected in duplicate in Encore samplers. A "bulk" sample is also routinely collected in a 125-mL jar fitted with Teflon-lined caps. The bulk sample can be used for direct purging by SW-846 Method 5030 or may be used to prepare the methnol extraction if the concentration exceeds the working range of the analytical system.

Soils collected in Encore samplers must be analyzed within 48 hours of collection or must be transferred within 48 hours to a sealed vial containing sodium bisulfate solution to preserve the sample. The hold time of the preserved sample is 14 days from the date of collection. The procedure for preparing soil samples is given in Section 9.2.

5.3 High level soil and waste samples are collected in glass containers (usually 125-mL clear glass) equipped with Teflon-lined caps. Soil samples may also be submitted as core samples contained in metal or plastic "tubes" or in 40-mL VOA vials. The samples are iced at the time of collection and stored at 4C+/-2C. The holding time for soil and waste samples is 14 days from date of collection.

5.4 TCLP leachate samples are collected with no headspace in Tedlar bags or syringe. The leachate samples are acidified at the time of collection (after the leaching procedure) with about 0.10mL of concentrated HCl per 40mL of sample and stored at 4C +/- 2C from the time leaching is completed until the analysis. The acidified leachate sample must be analyzed within 14 days of the leaching procedure If the sample is not acidified, the leachate must be analyzed within 7 days of the leaching procedure.

NOTE: Samples that are suspected of having very high concentrations of VOC should be segregated from the "routine" samples and stored in a manner that will minimize sample and laboratory contamination. See SL-SOP AN70. If possible, keep the field QC in the same storage refrigerator as the samples.

6.0 APPARATUS AND MATERIALS

The apparatus and materials listed in this section may vary from lab to lab. The items listed are to give guidance and to provide a general overview of the equipment employed in this analysis.

6.1 Mass spectrometer: HP5970, HP5971, or HP5972 equipped with a capillary direct interface

6.2 Gas chromatograph: HP5890 or equivalent GC equipped with a molecular jet separator

6.3 Purge and trap device

6.3.1 Tekmar 3000 Liquid Concentrator or equivalent

6.3.2 Autosampler: Tekmar 2016 or equivalent

6.3.3 Purging chamber: sparging vessel with purge tubes appropriate to the matrix and volume or weight of sample used.

6.3.4 Tekmar 3-ring trap, or equivalent, containing charcoal, Tenax, and silica gel. Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

6.4 Archon soil analyzer for low level soils, compatible with Tekmar purge and trap instruments. The instrument must be capable of automatically adding water and internal standard to the container while maintaining the septum seal, heating the sample to 40C, and spinning the stir bar to mix the sample during the purging step.

6.5 Data System compatible with the analytical system

6.6 Microsyringes: 10ul, 25ul, 50ul, 100ul, 250ul, 500ul, 2.5mL

6.7 Gastight syringe: 5mL, 25mL with luerlock tip

6.8 Volumetric flasks: 1.0mL, 10mL, 100mL

6.9 Recommended Columns

6.9.1 Restek 502.2: 60m x 0.53mm ID, 3.0um film

6.9.2 J&W DB-624: 75m x 0.53mm ID, 3.0um film

7.0 REAGENTS

Reagents must be tracked in accordance with SL SOP AN44:*Reagent Traceability*.

7.1 Reagent water-free of volatile contaminants (obtained by purging with inert gas or carbon filtration)

7.2 Methanol-Burdich and Jackson, Purge and Trap grade

7.3 Sodium bisulfate-reagent grade. This salt is hydroscopic and should be stored in a dessicator.

7.4 Soil preservation solution- Slowly add, while stirring, 200g of sodium bisulfate to a 1.0-L volumetric containing about 700mL of reagent water. After the salt has dissolved, dilute to volume with reagent water, transfer to a storage container, and store the solution in an area free from VOC-especially water soluble solvents such as acetone.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all stock solutions or neat compounds. All preparation steps must be in accordance with SL SOP AN41: *Standard Material Traceability*.

8.1 Preparation of Calibration Standards

8.1.1 Preparation of **Stock Standards** from Neat Compounds

The lab should attempt to obtain a certified primary standard or secondary standard before preparing stock standards from neat materials. If primary stock standards must be prepared in-house, the target concentration range is from 2000ug/mL to 10000ug/mL. SL-SOP AN43: *Standard Preparation* gives the general instructions for the preparation of the stock solutions from neat materials.

8.1.2 Preparation of the **Working Standard** from Stock Standards

The working standard is prepared from the primary stock standards that are either prepared from neat compounds or purchased as certified solutions. The working standard contains one or more of the target compounds at a concentration suitable for preparing the calibration standards, generally 10-200ug/mL. A known volume of the working standard is then added to a known volume of reagent water to make the calibration standard.

$$Cs \otimes Vs = Cws \otimes Vws$$

The volume of stock standard needed to prepare a working standard is given by the following equation:

$$Vs = \frac{Cws \otimes Vws}{Cs}$$

where

Cs = concentration of stock standard(ug/mL)
Vs = volume of stock standard needed (mL)
Cws = concentration of the working standard(ug/mL)
Vws = volume of working standard to be prepared (mL)

EXAMPLE

Prepare 1.0mL of a 25ug/mL working standard from a 2000ug/mL stock standard.

$$Vs = \frac{Cws \otimes Vws}{Cs} = \frac{25ug/mL \otimes 1.0mL}{2000ug/mL} = 0.0125mL = 12.5uL$$

12.5uL of the 2000ug/mL stock standard is diluted to 1.0mL with methanol. The concentration of the working standard is 25ug/mL.

The standards and standard concentrations listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document the standard preparation procedures in the standard traceability log.

8.1.3 Preparation of the **Calibration Standards** from the Working Standards

The calibration standards are the standards that are analyzed on the instrument. The calibration standard is made by adding a known volume of the working standard to a known volume of reagent water. The instrument must be calibrated using a minimum of five calibration standards. The lowest level standard must be at the reporting limit and the rest of the standards will define the working range of the analytical system.

8.1.3.1 Add 5.0mL of reagent water to a 5mL glass syringe or 25ml of reagent water to a 25-ml glass syringe.

8.1.3.2 Add a known volume of the working standard (the following table or Vws below) to 5.0mL or 25ml of reagent water.

$$Vws = \frac{Ccal \otimes Vcal}{Cws}$$

where

Ccal = concentration of the calibration standard(ug/L)
Vcal = volume of the calibration standard to prepare(L)-0.0050L or 0.0250L
Cws = concentration of the working (secondary) standard(ug/mL)
Vws = volume of the working standard required to prepare the calibration standard(mL)

NOTE: The calibration standards for the Low level soils are prepared using the same procedures as for the 5mL water purge except that the standards are purged at 40C. The lab has the option of using blank sand in the calibration standards.

The calibration standards listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document these standard preparation procedures in the standard traceablity log.

9.0 SAMPLE PREPARATION

Composite samples can be prepared using the guidance provided in SL-SOP AN70.

9.1 **Aqueous samples** are analyzed directly by purge and trap/GC-MS. No sample preparation is necessary except to homogenize the sample prior to subsampling. The pH of liquid samples is checked and recorded prior to analysis to determine if the sample has been properly preserved.

9.2 Preparation of Soil Samples (5035)

9.2.1 Remove the Encore samples and the bulk sample from the storage area.

9.2.2 Test an aliquot of the bulk sample for the presence of carbonates.

- Transfer 5g of sample from the bulk sample to a 40mL vial..
- Add 5ml of the sodium bisulfate solution and shake the vial .
- If the sample exhibits effervescence, the Encore samples should be preserved as described above using 5mL of volatile-free water in place of the sodium bisulfate solution and placed in a freezer at –10C. The analytical hold time for frozen samples is 14 days from collection.
- If no effervescence is noted, the Encore samples may be preserved with 5mL soil preservation solution.

9.2.3 Add a stir bar to a vial and weigh the vial and record its tare weight(or tare the vial and stir bar weight by pressing the autotare button).

9.2.4 Transfer the sample from the Encore sampler to the tared vial and record the weight of the sample log.

If the sample effervesced during the carbonate test (9.2.2), add 5.0mL of reagent water and freeze at –10C. The hold time is 14 days from collection

If not, add 5.0mL of the soil preservation solution, seal the vial, and store the sample at 4C until the time of analysis. The preserved sample must be analyzed within 14 days of collection.

NOTE: A preparation blank is prepared when Encore samples are transferred. The preparation blank contains the same reagents as the samples-either 5mL of reagent water or 5mL of soil preservation solution.

9.3 A methanol extraction is prepared when the concentration of the target compounds (by direct purge) exceeds the working range of the calibration curve. The bulk sample, collected in the 125-mL sample container, is used to prepare the methanol extraction. Carry out the preparation quickly to minimize the loss of volatiles.

-Mix the sample with a stainless steel spatula and transfer 5g (+/- 0.5g) to a glass vial.

-Add 4uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 5.0mL of purge and trap grade methanol. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 5.0g and 100% percent solids, is calculated:

$$Ct(ug/kg,dw) = \frac{0.004mL \otimes 2500ug/mL}{0.0050g \otimes solids} = 2000ug/kg,dw$$

-Shake the sample for one minute. Allow the solvent to separate from the solids portion of the sample and transfer a 1-2mL aliquot of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

-For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 4uL of the surrogate spiking solution to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 125uL of the extract.

The lab control standard is prepared by adding 4uL of the surrogate spiking solution and 4uL of the matrix spiking solution to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 125uL of the extract.

The matrix spikes are prepared by adding 4uL of the surrogate spiking solution (2500ug/mL) and 4uL of the matrix spiking solution (2500ug/mL) to 5g aliquots of the sample selected for the MS/MSD. Quickly add 5.0mL of purge and trap grade methanol to each sample and shake for one minute. Analyze 125uL of the extract or a smaller volume if the VOC concentration is high.

-Add 125uL of the extract (or a smaller volume if the VOC concentration exceeds the linear range of the system with 125uL) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

9.4 Methanol Extraction for Wastes

Carry out the preparation quickly to minimize the loss of volatiles.

9.4.1 Mix the sample with a stainless steel spatula and transfer 1g (+/- 0.2g) to a glass vial.

9.4.2 Add 10uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. If the sample is completely soluble in the methanol, dilute to a final volume of 10mL The theoretical concentration of the surrogates in the sample, assuming a sample weight of 1.0g , is calculated:

$$Ct(ug/kg) = \frac{0.010mL \otimes 2500ug/mL}{0.0010g \otimes solids} = 25000ug/kg$$

9.4.2 Shake the sample for one minute. Allow the solvent to separate from the solids portion of the sample and transfer a 1-2mL aliquot of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) to 10mL of purge and trap grade methanol. Assume a sample weight of 1.0g. Analyze 100uL of the extract.

The lab control standard is prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 125uL of the extract.

The matrix spikes are prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 1g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for one minute.

Add 100uL of the extract (or a smaller volume) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

NOTE: Waste samples will usually require significant dilution prior to analysis.

10.0 PROCEDURE

The following instrument conditions are recommended. The actual conditions may vary due to differences in instrumentation. The lab must document the instrument conditions in the maintenance log, the data system, or on the analysis log.

10.1 Instrument Conditions

10.1.1 GC Conditions
GC conditions may vary according to the environment and condition of each instrument. The lab must document the instrument conditions to assure consistent results and to aid in trouble-shooting the analytical system. Each lab is responsible for assuring that the conditions necessary to achieve adequate separation and sensitivity of the target analytes are maintained.

10.1.1.1 Example GC temperature program(ambient cooling)

Initial column temperature: 35 C for 3 minutes
Column temperature program 1: 20C per minute
Intermediate column temperature: 70C for 4 minutes
Column temperature program 2: 10C per minute
Final column temperature: 200C for 5.25 minutes

10.1.1.2 Column flow: Approximately 5-10mL/minute helium with a make-up of 20-25mL/minute helium. Total flow into the jet separator should be about 30mL/minute. The vacuum gauge on the jet separator will read about 0.5Torr.

10.1.1.3 Mass Spectrometer and interface parameters

Jet separator temperature: 240C
Mass spectrometer interface: 240C
Mass spectrometer source temperature: factory set at 300C
range: 35-300amu, with a minimum scan cycle of 1 scan per second

10.1.2 Purge and Trap Conditions
The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

10.1.2.1 "Three ring trap"-charcoal, Tenax, silica gel
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 180C
Bake time: 8 minutes at 225C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.1.2.1 VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 225C
Bake time: 8 minutes at 250C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be Low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water soluble targets-ketones, ethers, bromoform-may be low and the reporting limit may not be achieved on a routine basis.

10.2 Calibration

10.2.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each 12-hour clock as a check on the "**tune**" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage. If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

10.2.2 Evaluate the 4-BFB peak.

10.2.2.1 The chromatogram should exhibit acceptable baseline behavior and the 4-BFB peak should be symmetrical. A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

10.2.2.2 The spectrum of the 4-BFB must meet the criteria listed in the attached SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 5 scans from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.

10.2.2.3 The following records must be kept for each 4-BFB analysis that meets the criteria:
- the date, time, and data file of the analysis
- a spectrum of the scan or averaged scans
- a tabulation of the ion abundances of the scan

10.2.2.4 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , or other anomalies.

10.2.2.5 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

10.2.3 After the 4-BFB criteria has been met, the initial calibration standards are analyzed

10.2.3.1 Prepare the initial calibration standards. The lab must document the "recipe" used to prepare the calibration standards. An example of a standard preparation is given in Section 8.

10.2.3.1.1 Remove the plunger from the syringe and fill the barrel to overflowing with reagent water (syringe valve in the "red" position).

10.2.3.1.2 Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL or 25mL)

10.2.3.1.3 Briefly remove the syringe valve and inject the standards and internal standards into the syringe.

NOTE: Use the internal standard (IST) mix when preparing the calibration standards for analysis. The surrogates are already included in the standard mixes.

10.2.3.2 Load the standard(s) onto the purge and trap device and begin the analysis. All pertinent information concerning the standards must be recorded on the analysis log. The standards must be clearly identified and traceable to the preparation steps.

NOTE: The standards for low level soil samples are prepared in the same manner as the 5mL standards. The standards for the low level soils are purged at 40C. The lab has the option of using blank sand or soil in the calibration standards and the blank in the low level soil analysis.

10.2.3.3 After the acquisition has taken place, evaluate the calibration standards to ensure that each target compound, surrogate, and internal standard has been correctly identified. The analyst must be careful to complete this step before proceeding.

10.2.3.4 After each target compound, surrogate, and internal standard has been correctly identified, the relative response factor for each target compound and surrogate is calculated using the data system or using a PC spreadsheet as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where
Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (see the attached quantitation report for a list of the compounds that are associated with the various internal standards)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug on-column)
Cis = concentration or mass on-column of the internal standard (ug/L or ug/kg OR ng or ug on-column)

10.2.3.5 The average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$RRFavg = \frac{RRF1 + RRF2 + ... + RRFn}{n}$$

where n = number of calibration levels

10.2.3.6 Calculate the standard deviation (SD) for the target compounds and surrogates at all calibration levels:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(RFi - RFavg)^2}{n-1}}$$

where
Rfi = response factor of a target compound in the individual calibration level
Rfavg = average response factor
n= number of calibration levels

10.2.3.7 Calculate the relative standard deviation (% RSD) of the calibration levels for each target:

$$\%RSD = \frac{standard\ deviation}{RRFavg} \otimes 100$$

10.2.3.8 The results of the initial calibration are evaluated against the Calibration Check Compound(CCC) criteria and the System Performance Check Compound (SPCC) criteria. The CCC and SPCC criteria are listed below. The CCC and SPCC criteria must be met before samples can be analyzed.

Calibration Check Compounds – CCC Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
Less than 30% RSD	<20% difference from initial calibration

System Performance Check Compounds-SPCC

SPCC	5g/5mL minimum RRF	25mL minimum RRF
Chloromethane	0.10	0.10
1,1-Dichloroethane	0.10	0.10
Chlorobenzene	0.30	0.10
Bromoform	>0.10	>0.10
1,1,2,2-Tetrachloroethane	0.30	0.10

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

The results for all target compounds are evaluated for linearity. If the %RSD is less than 15%, the calibration curve is assumed linear through the origin and the average response factor can be used for quantitation. If the average response factor for the target exceeds 15% (including any CCC), the analyst must use the calibration curve option.

NOTE: The lab has the option of using a calibration curve for all analytes. This is the recommended course of action-especially for data systems that cannot automatically calculate the relative response factors.

10.2.4 The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug)
Cis = concentration of the internal standard (ug/L or ug/kg OR ng or ug)

A regression curve may be used to define the concentration/response relationship. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPs for other exceptions to using non-linear curve fitting.

10.2.5 After the calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the SL CQAP or other specified QAP.

10.2.5.1 If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compounds must meet the criteria.

10.2.5.2 If the concentration of any target compound is above the RL in Table 5 of the SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of samples can take place.

If the method blank repeatedly fails to meet the criteria, contact the immediate supervisor to determine the cause of the problem and to determine a course of action. This action may include re-cleaning the sparging tubes (with soap, hot water, and methanol), purging the effected autosampler ports with heated methanol, flushing the purge and trap ALS concentrator with methanol, replacing the trap, changing the transfer line, and changing the column. A method blank is then analyzed after taking the corrective action to demonstrate that the contamination has been eliminated. Once the system is determined to be free from contamination, sample analysis may begin. Method blanks may be required after the analysis of samples that contain very high levels of VOC.

10.3 Continuing Calibration Verification

10.3.1 At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of 50ng of 4-BFB. This criteria must be met before the analysis of the calibration check standards can take place.

10.3.2 After the tune criteria has been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be a nominal concentration of 50ug/L-kg for 5ml/5g and 10ug/L for 25mL with ketones and poor purgeables at higher concentrations. The CCC and SPCC criteria must be met before the analysis of the method blank can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration
Cccv = measured concentration using the selected quantitation method

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If this criteria is not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed.

10.3.4 The calibration standard must also be evaluated for internal standard retention time and area.

10.3.4.1 If the retention time of any internal standard changes by more than 30 seconds from the last 12-hour calibration check, the analytical system must be inspected for problems and corrective action instituted.

10.3.4.2 If the extracted ion current profile (EICP) area for any of the internal standards changes by more than a factor of two (-50% to +100%) from the last calibration check standard, the analytical system must be inspected for problems and corrective action instituted.

10.3.5 After the continuing calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the SL CQAP.

10.3.5.1 If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compound must meet the criteria.

10.3.5.2 If the concentration of any target compound is above the RL in Table 5 of the SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of client samples can take place. Refer to Section 10.2.5.2 for suggested corrective action.

10.4 Sample Analysis

10.4.1 Aqueous Samples-5ml or 25mL sample
This method allows the use of either a 5mL sample volume or a 25ml sample volume. The analyst must use the same volume as was used for the calibration standards-if a 5mL sample is used, it must be quanted off of the 5mL calibration curve; if a 25ml sample is used, it must be quanted off of the 25mL calibration curve. Samples are analyzed only after the 4-BFB criteria, the initial or continuing calibration criteria has been met, and the method blank criteria has been met.
See the SOP Summary for the analytical sequence.

10.4.1.1 Remove the samples to be analyzed from the refrigerator and allow the samples to come to ambient temperature.

10.4.1.2 Put on a pair of gloves before transferring the sample from the vial to the syringe. The sample is most likely preserved with acid or may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. ***Gloves must be worn when handling samples.***

10.4.1.3 Mix the contents of the vial by inverting the vial several times. Check to see if there are air bubbles present in the sample. If air bubbles are present, use another vial if available. Make a note on the analysis log if the sample used contained bubbles and notify the supervisor and/or the project manager.

10.4.1.4 Remove the plunger from the glass syringe. Attach a syringe valve to the syringe Luer-tip to prevent sample from spilling out of the syringe when sample is added.

10.4.1.5 Open the vial of the well mixed sample and gently pour the sample into the syringe barrel. The sample should fill the barrel of the syringe and overflow to allow trapped air bubbles to escape.

10.4.1.6 Replace the plunger into the syringe barrel. Try not to let air bubbles get into the barrel. If air bubbles are present, turn the syringe up, open the syringe valve , and expel the air while adjusting the volume to 5.0mL or 25mL. If no air bubbles were trapped, adjust the syringe to volume.

NOTE: For TCLP leachate samples, use 1.25mL of sample (1:4 dilution).

10.4.1.7 Open the syringe valve and inject the internal standard/surrogate (ISSU) mix into the sample.

10.4.1.8 Transfer the sample from the syringe to the purge and trap device. Record all of the sample identification information on the analysis log. Check the pH of the sample with pH paper and record the pH on the instrument log or other appropriate log.

10.4.1.9 Analyze the samples using the purge and trap and GC/MS conditions used for the initial and continuing calibration standards.

10.4.1.10 Determine the concentration of the samples and QC items. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

10.4.1.10.1 A dilution is made when a volume of the sample is mixed with the reagent water to a final volume of 5.0mL or 25ml, depending on which curve is being used. The dilution reduces the concentration of the original sample to bring the concentration into the working range of the calibration curve.

10.4.1.10.2 The dilution factor is calculated by dividing the volume of sample into the volume used for the calibration curve.

$$DF = \frac{final\ volume\ of\ dilution(mL)}{volume\ of\ sample\ used(mL)}$$

For example, if 1.0mL of sample is diluted to final volume of 5.0mL, the dilution factor is 5. (5.0/1.0 = 5). If 1.0mL of sample is diluted to a final volume of 25mL, the dilution factor is 25 (25/1=25).

The following table gives some dilution factors:

Volume of Sample (mL)	Volume of Reagent Water (mL)	Final Volume (mL)	Dilution factor
5.0	0	5.0	1
2.5	2.5	5.0	2
1.0	4.0	5.0	5
0.5	4.5	5.0	10
0.10	4.9	5.0	50
25.0	0	25.0	1
5.0	20.0	25.0	5
2.5	22.5	25.0	10
1.0	24.0	25.0	25
0.50	24.5	25.0	50
0.10	24.9	25.0	250

NOTE: The same volume of internal standard/surrogate mix (ISSU) is added to the dilution as was added to the undiluted sample.

10.4.2 Low Level Soil Samples by Heated Purge and Trap (Method 5030)

This method allows the use of 1g to 5g aliquot of the subsample. The well mixed soil sample is combined with 5mL of water containing the internal standard and surrogates in a soil sparging vessel. The sample is purged at 40C. The working calibration range of the low level procedure is nominally 5ug/kg to 1000ug/kg. Samples are analyzed only after the 4-BFB criteria, the initial or continuing calibration criteria has been met, and the method blank criteria has been met

10.4.2.1 Remove the samples to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.4.2.2 Put on a pair of gloves before transferring the sample from the vial to the sparging vessel. The sample may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. ***Gloves must be worn when handling samples.***

10.4.2.3 Mix the contents of the container by stirring the sample with a spatula. The sample must be homogenized before an aliquot is weighed into the sparging vessel. Weigh 1g to 5g of the sample into the sparging vessel. Attach the sparging vessel to the autosampler. Record the weight of sample, to the nearest 0.1g, on the analysis log. Do not use less than 1g of sample for the low level soils.

10.4.2.4 Remove the plunger from the glass syringe. Attach a syringe valve to the syringe Luer-tip to prevent reagent water from spilling out of the syringe. Add reagent water to the syringe and adjust the volume to 5.0mL.

10.4.2.5 Open the syringe valve and inject the internal standard/surrogate (ISSU) mix into the reagent water. Add the reagent water with the ISSU mix to the sparging vessel.

10.4.2.6 Attach the heater jacket or other heating device to the sparging vessel and analyze the samples using the same conditions used for the initial and continuing calibration standards-purge temperature for low level soils is 40C. Record the sample information on the analysis log.

10.4.2.7 Determine the concentration of the samples and QC items. If the concentration of a target compound is above the highest calibration standard, a smaller sample aliquot is weighed and analyzed. Do not use less than 1g of sample for the low level soils. If the concentration of a target compound exceeds the linear range using a 1-g aliquot of soil, the medium level soil extraction must be used.

Sample Size	Nominal low range for concentration*	Nominal High range for concentration*
5.0g	5ug/kg	200ug/kg
1.0g	25ug/kg	1000ug/kg
MeOH extractions		
5g to 10mL- 0.10mL of extract	500ug/kg	20000ug/kg
5g to 10mL- 0.050mL of extract	1000ug/kg	40000ug/kg
5g to 10mL- 0.025mL of extract	2000ug/kg	80000ug/kg
5g to 10mL- 0.010mL of extract	5000ug/kg	200000ug/kg

* the nominal concentration is based on a 25ng on-column quantitation. Compounds with higher quantitation limits will be proportionally higher.

10.4.3 Low Level Soil Samples by Heated Purge and Trap (Method 5035)

10.4.3.1 Remove the samples to be analyzed from the refrigerator and allow the sample to come to ambient temperature. Inspect the vial for cracks or obvious breaches in the septum.

10.4.3.2 Weigh the sample on a top-loading balance and record the weight in the sample logbook. Calculate the weight of sample by subtracting the tare weight of the container from the weight of the sample and container:

$$Weight(g) = W2 - W1$$

where
W2 = weight of container, sample, stir bar, and preservative(g)
W1 = tare weight of the container, stir bar, and preservative(g)

Be sure to account for the weight of extraneous materials affixed to the container.

10.4.3.3 Load the samples on to the soil purging unit. Standards and QC items must be analyzed under the same heated purge and trap conditions.

10.5.3 Analysis of Methanol Extracts of Soils and Wastes

The methanol extraction is used when the concentration of one or more target compounds exceeds the linear range of the low level purge technique (>1000ug/kg). Samples are analyzed only after the 4-BFB criteria, the initial or continuing calibration criteria has been met, and the method blank criteria has been met. Medium level soil extracts are quanted using the ambient purge calibration curve. Sample preparation steps are included in Section 9.

10.5.3.1 Remove the plunger from the 5.0-mL syringe and fill the barrel to overflowing with reagent water(syringe valve in the "red" position).

10.5.3.2 Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL)

10.5.3.3 Briefly remove the syringe valve and inject the sample extract(maximum of 100uL) and 5uL of the internal standard (IST) solution into the syringe.

NOTE: Use the internal standard(IST) mix when preparing the medium level samples. Recall that the surrogates have already been added to the sample prior to methanol extraction.

10.5.3.4 Load the sample on to the purge and trap device and begin the analysis. All pertinent information concerning the samples must be recorded on the analysis log. The samples must be clearly identified and traceable to the extraction log. These conditions must be the same as was used for the initial and continuing calibration standards-ambient purge for aqueous samples.

10.5.3.5 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, a smaller aliquot of the methanol extract is reanalyzed.

NOTE: It is possible to dilute the surrogates in the sample extract below the linear range of the calibration curve. The minimum extract aliquot that can be used to provide a quantifiable result for the surrogates and matrix spikes is 0.020mL (20uL). It is not probable that the recovery of the surrogates would be 100%, which is necessary to provide a quantifiable result using a 0.010mL(10uL) aliquot of the extract.

mL Methanol Extract-5g to 10mL MeOH	Surrogates- theoretical ng on-column
0.100	250
0.050	125
0.020	50
0.010	25-quantitation limit
0.0050	12.5-below the quantitation limit

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

11.1.1 Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the daily calibration standard or a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to positively identify a compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{\textit{retention time of the target compound}}{\textit{retention time of the associated internal standard}}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. These ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification. Tentative identifications of non-targets will be made only by analysts having completed the training specified in the training schedule.

11.1.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.2.2 The relative intensities of the major ions should agree within +/-30%.

11.1.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.1.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.1.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.1.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult other analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.1.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculation is

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes DF$$

where
Cis = concentration of the internal standard, ug/L
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
DF = dilution factor

Soils by Heated P/T

$$TIC\ (ug/kg, dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{5.0g}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
W = weight of sample analyzed, g
solids = decimal equivalent of percent solids

Soils by Methanol Extraction

$$TIC\ (ug/kg, dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{Vcal}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = decimal equivalent of the percent solids(percent solids/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

where
Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

11.2 Calculations for Samples-Internal Standard Technique

11.2.1 Aqueous Samples

11.2.1.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes DF$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
DF = dilution factor

11.2.1.2 If a regression curve is used, the concentration is given:

$$concentration(ug/L) = concentration(curve) \otimes DF$$

where
DF = dilution factor

11.2.1.3 The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes DF$$

where
DF = dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

11.2.2 Soils by Heated P/T

11.2.2.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg, dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{5.0g}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/kg)
RRFavg = average response factor of the compound being measured
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

11.2.2.2 If the regression curve is used, the concentration is given:

$$conc(ug/kg, dw) = Ccurve(ug/kg) \otimes \frac{5.0g}{(W)(solids)}$$

where
Ccurve = concentration from curve(ug/kg)
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

11.2.2.3 The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The SL CQAP assumes W= 5.0g and solids = 1.

11.2.3 Methanol Extraction Soils and Wastes

11.2.3.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg, dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{Vcal}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = (percent solids)/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

11.2.3.2 If the regression curve is used, the concentration is given:

$$conc(ug,kg,dw) = Ccurve(ug/L) \otimes \frac{Vcal}{(W)(solids)}$$

where
Vcal = volume that calibration curve is based on (0.005L or 0.025L)
W = weight of sample added to the reagent water (g)-defined above(11.2.3.1)

11.2.3.3 The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the reagent water (g)
solids = (percent solids)/100)

The SL CQAP assumes W= 5.0g and solids = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. Note that the method blank for liquid samples and low level soils is clock-specific and that the method blank for medium level soil samples is batch-specific.

SL-SOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. This criteria is summarized in the attached 8260 SOP Summary.

SL SOP AN02 also contains the calculations for accuracy and presicion and the calculations for the theoretical concentrations of surrogates, lab spikes, and matrix spikes.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration is the preparation and analysis of spiked reagent water. Section 8.3 of EPA Method 8260A gives the general procedure for the performance of the IDOC and Table 6 of EPA Method 8260A gives the acceptance criteria for the accuracy and precision.

12.3 Method Detection Limit

The method detection limit is determined in accordance with SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. Section 10 of the CQAPs contain guidance on preventive maintenance.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time. See instrument manufacturers' manuals for guidance on locating and repairing instrument problems.

15.0 REFERENCES

1. *Savannah Laboratories' Comprehensive Quality Assurance Plan* and *Savannah Laboratories' Corporate Quality Assurance Plan*, current revisions.

2. Method 8260B. *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846.* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

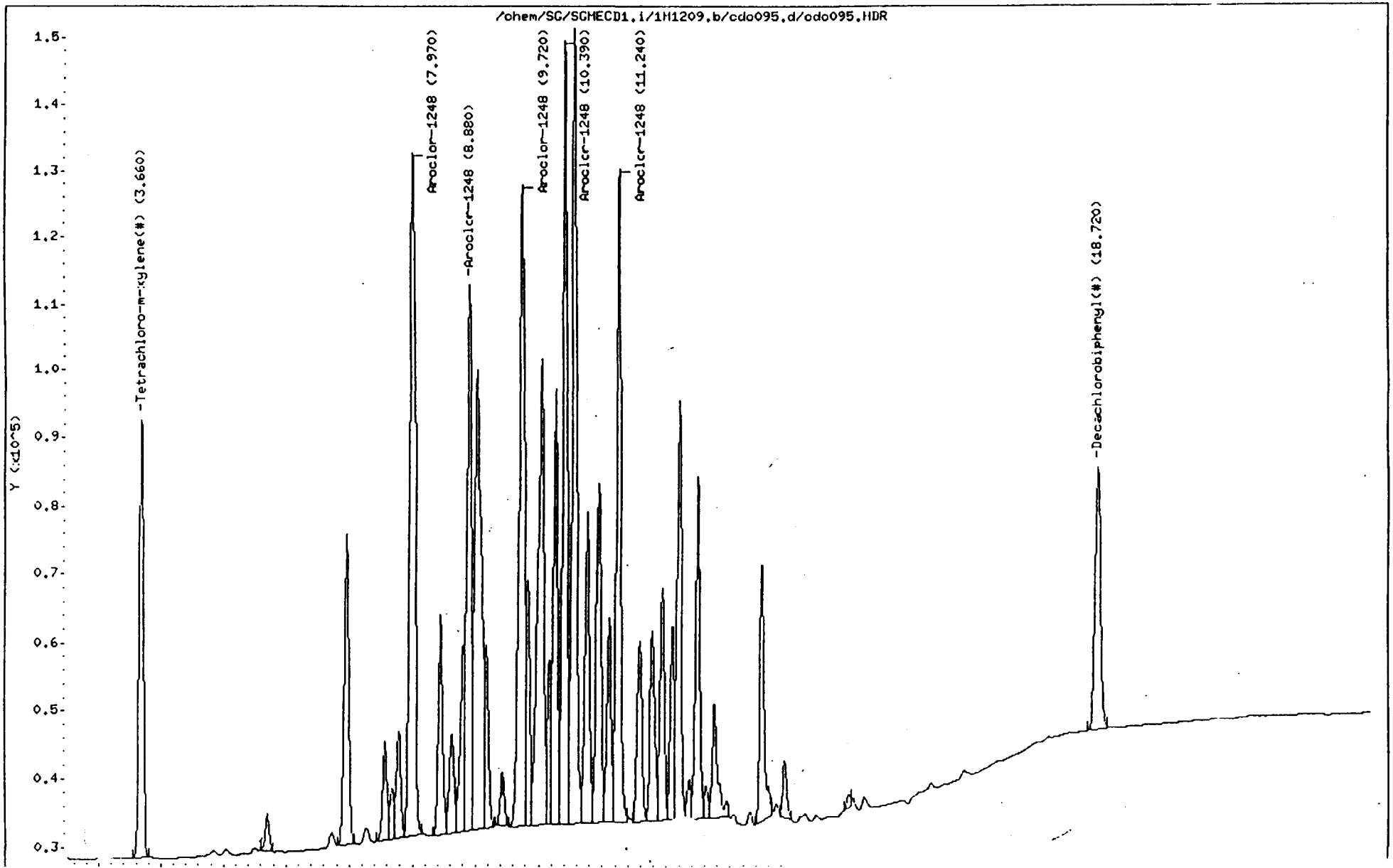
Data File: /chem/SG/SGHECD1.i/1H1209.b/cdo095.d
Date : 09-DEC-97 21:13:00
Client ID: AR1248-04
Sample Info: AR1248-04~1H1209

Instrument: SGHECD1.i

Operator: BW

Column phase: DB-608

Column diameter: 0.53



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Date : 09-DEC-97 21:40:00

Client ID: AR1254-04

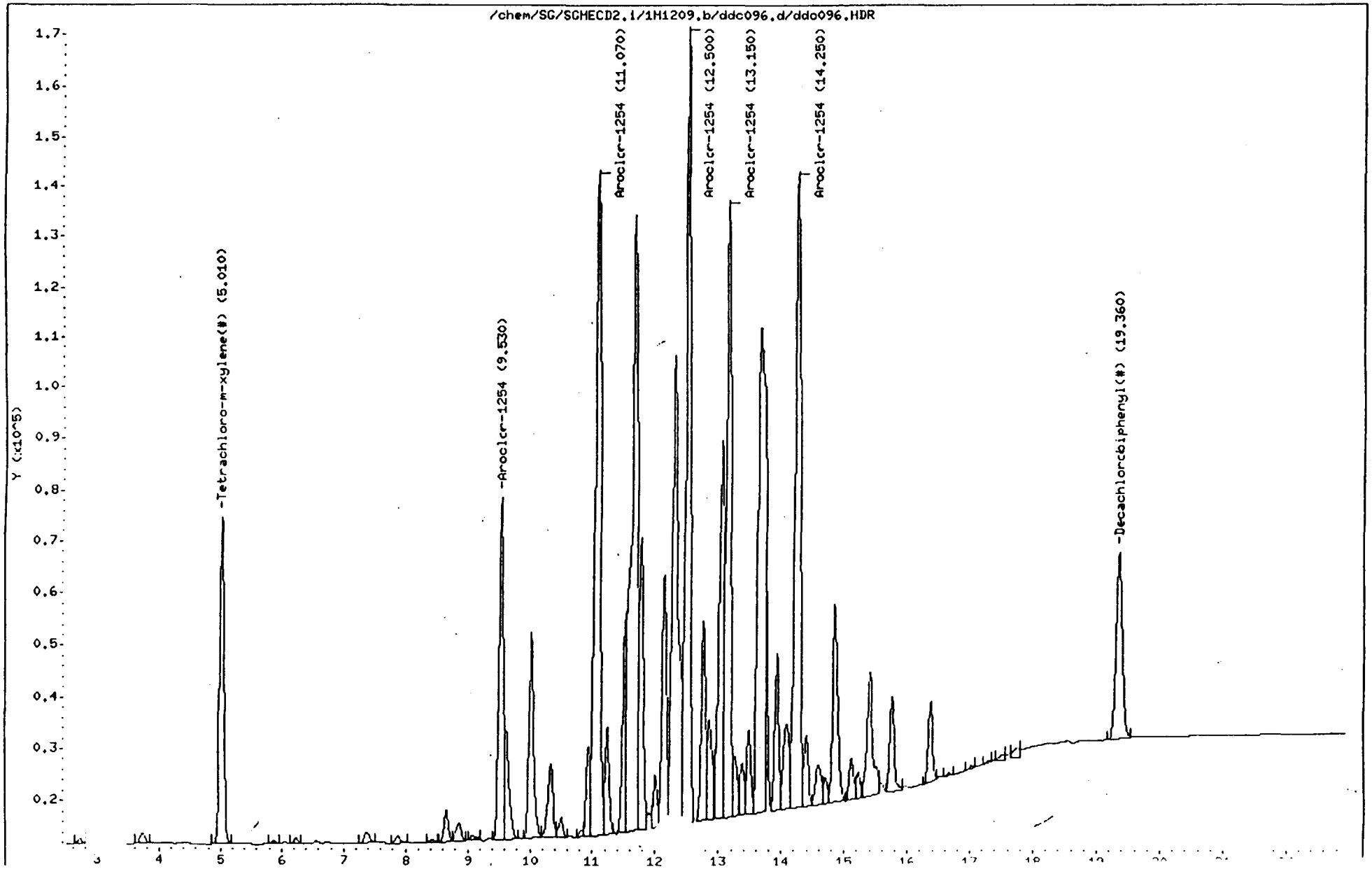
Sample Info: AR1254-04~1H1209

Column phase: DB-5

Instrument: SGHECD2.i

Operator: BW

Column diameter: 0.53



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Date : 09-DEC-97 21:40:00

Client ID: AR1254-04

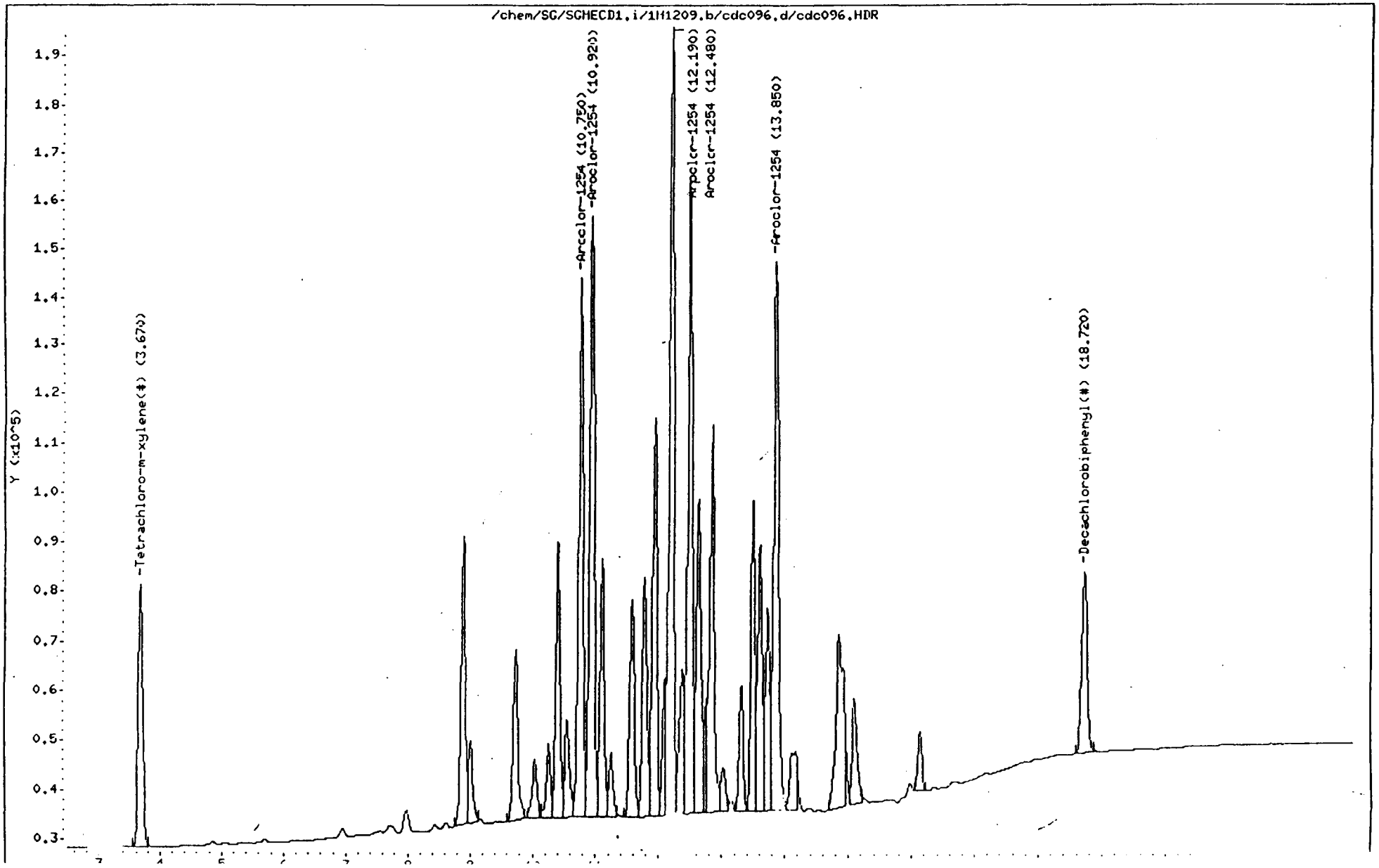
Sample Info: AR1254-04~1M1209

Column phase: DB-608

Instrument: SGHECD1.i

Operator: BW

Column diameter: 0.53



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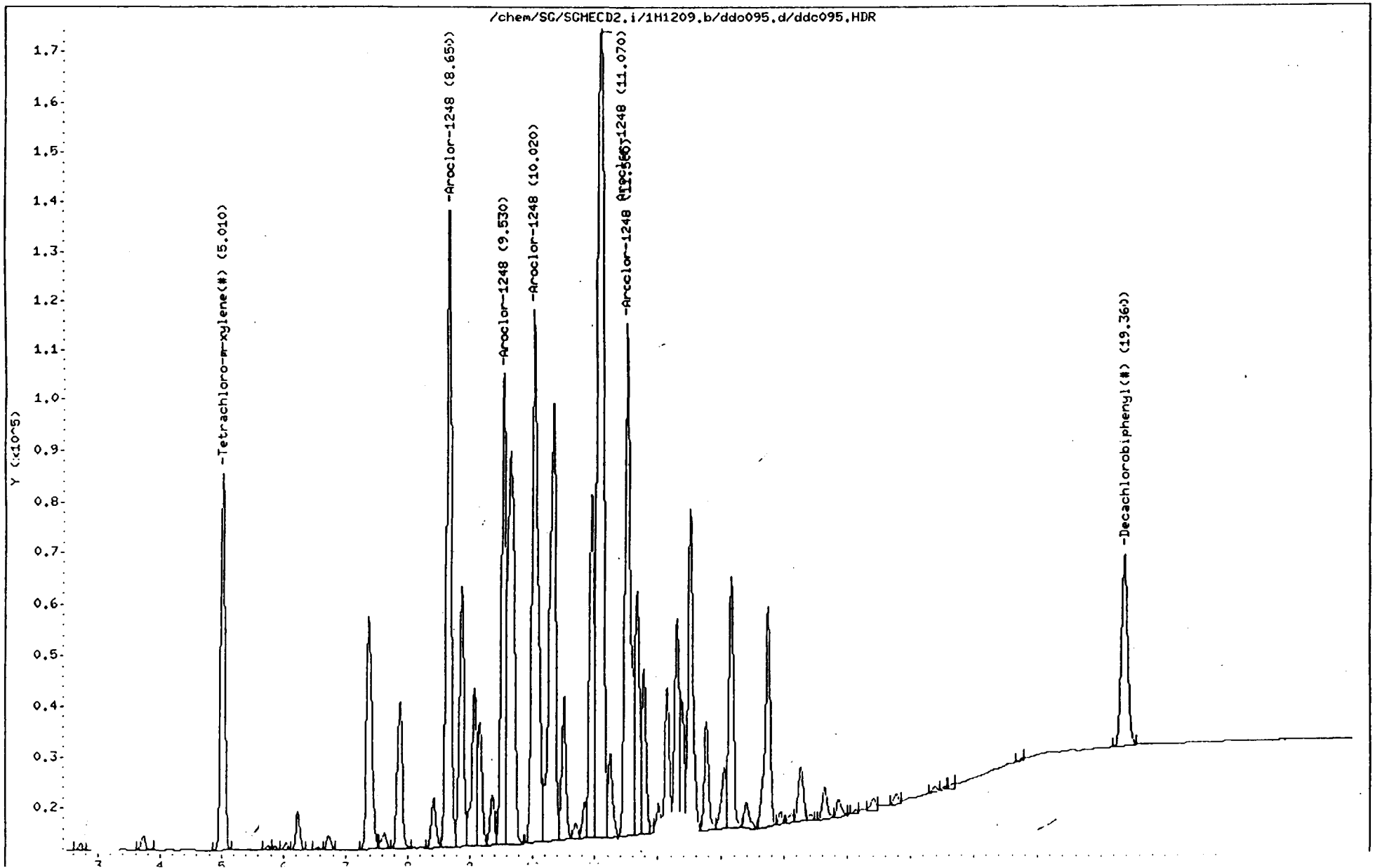
Date : 09-DEC-97 21:13:00
Client ID: AR1248-04
Sample Info: AR1248-04~1H1209

Instrument: SGHECD2.i

Operator: BW
Column phase: DB-5
Column diameter: 0.53



Data File: /chem/SG/SGHECD1.i/1H1209.b/cdc097.d

Date : 09-DEC-97 22:08:00

Client ID: AR1268-04

Sample Info: AR1268-04~1H1209

Column phase: DB-608

Instrument: SGHECD1.i

Operator: BW

Column diameter: 0.53

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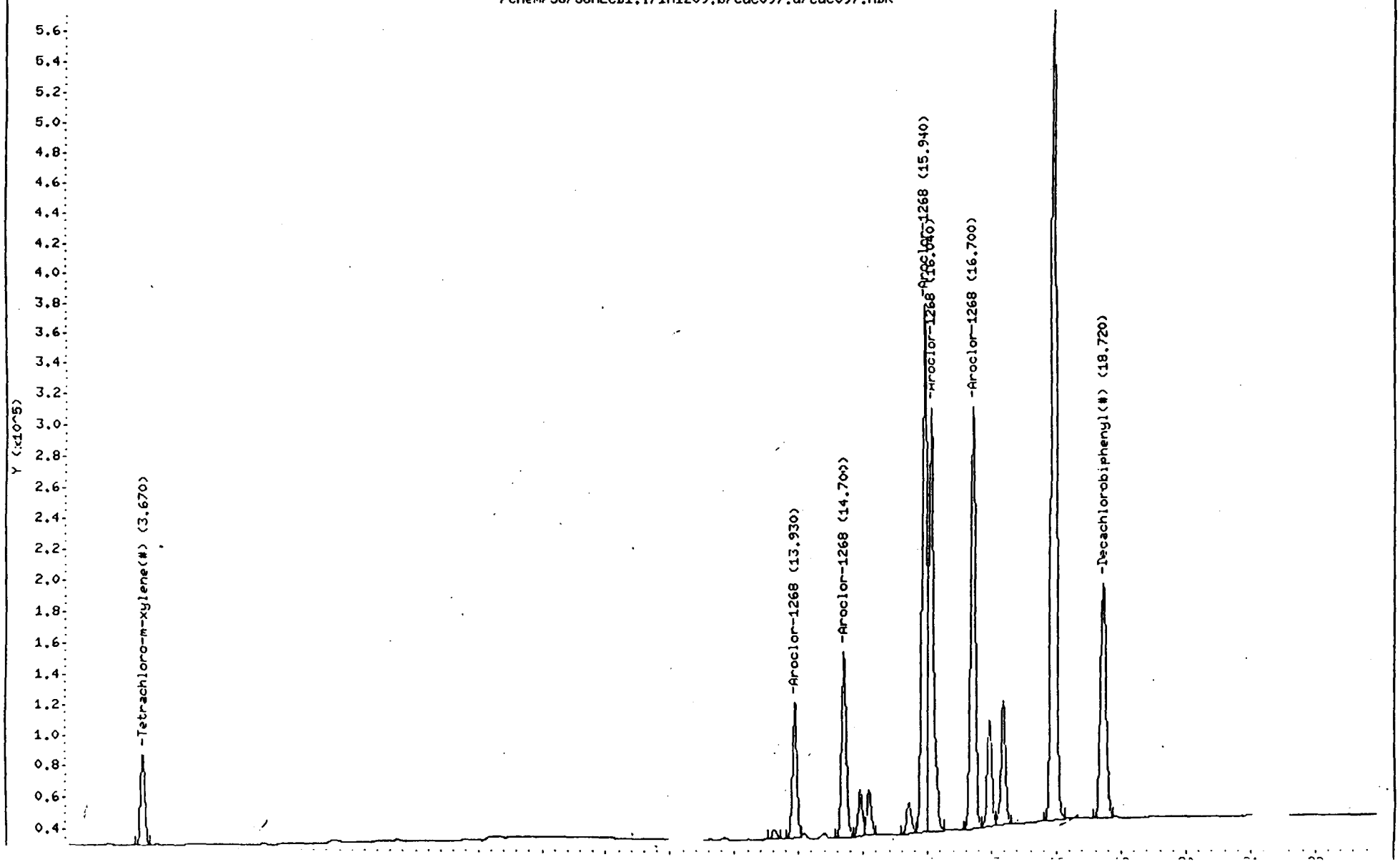

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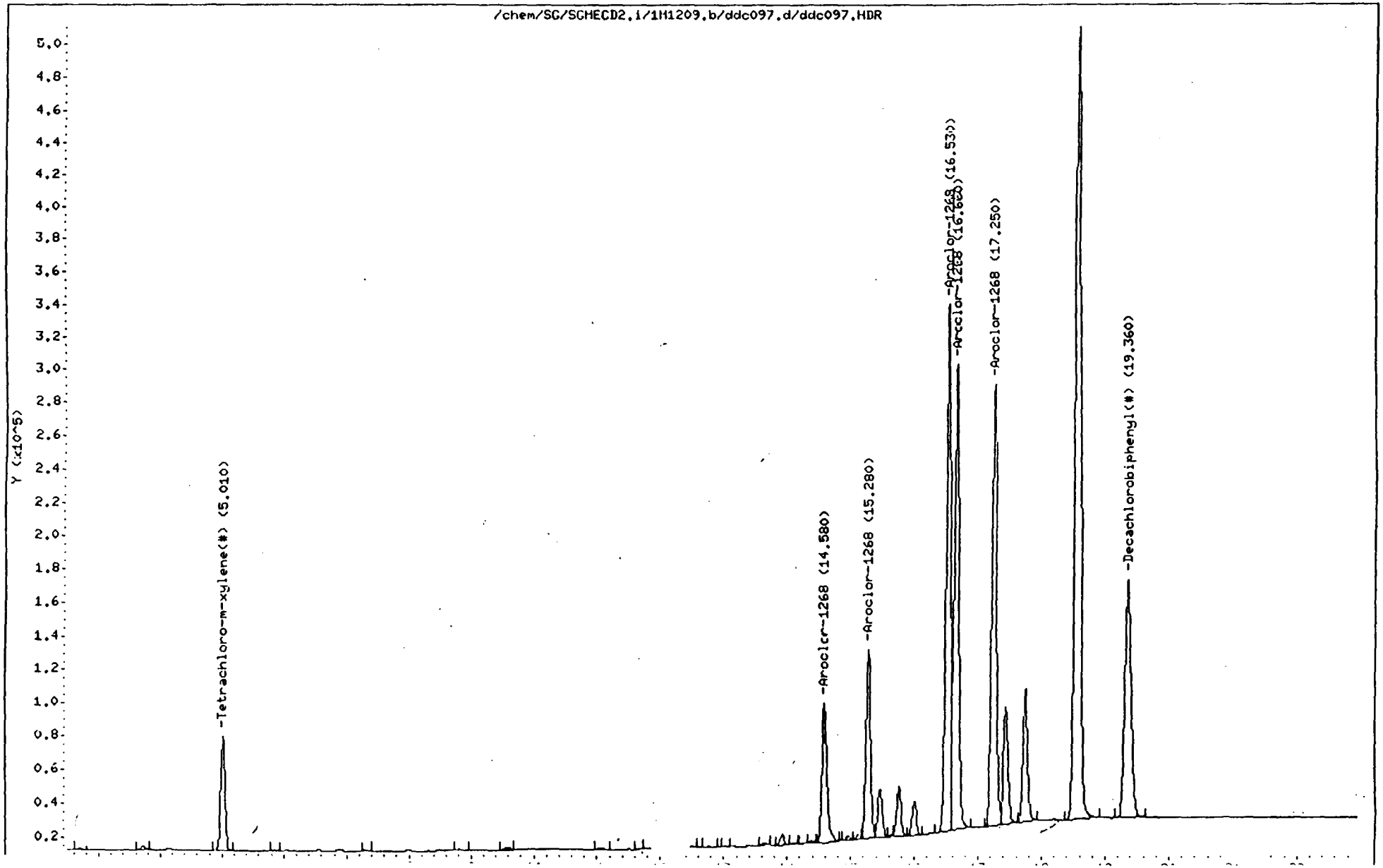
Sample Info: AR1268-04~1H1209

Instrument: SGHECD2.i

Operator: BW
Column phase: DB-5
Column diameter: 0.53



Appendix A

VOLATILES BY GC/MS WORKING STANDARDS -EXAMPLE

These standards can be used to prepare the working standards for EPA Method 8260 to report the TCL (target compound list) compounds and the extended list of target compounds generally associated with EPA 8260. The standards are prepared in purge and trap grade methanol and are stored at 4C with minimum headspace.

Working Standard 1 (TCL WS-1)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #2	2000	12.5	25
VOA Cal #3	2000	12.5	25
VOA Cal #4	2000	12.5	25
1,2,-DCB	5000	5.0	25
1,3-DCB	5000	5.0	25
1,4-DCB	5000	5.0	25
2-CEVE	1000	125	125

Working Standard 2 (TCL WS-2)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #1	5000	25	125
8260 Surrogates	2500	10	25

Working Standard for GASES (TCL GASES)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
502.2 Cal 1	2000	12.5	25

Appendix A

Working Standard 3 (8260 WS-3)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
8260 Custom Mix #1	200	125	25
8260 Custom Mix #2	200	125	25
1,1,2,2-Tetrachloroethane	2000	12.5	25

Appendix A

Internal Standard (8260 ISTD)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50

Internal Standard/Surrogate (8260 ISSU)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50
8260 Surrogate	2500	20	50

Tune Evaluation Standard (4-BFB)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
4-BFB	5000	10	50

Matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
Matrix Spiking Solution	2500	20	50

TCLP matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
TCLP Spiking Solution	2000	16	125

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS - EXAMPLES

The following calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	TCL-1 *	TCL-2 *	TCL-3 *	TCL-4 *	TCL-5 *	TCL-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	8260-1 *	8260-2 *	8260-3 *	8260-4 *	8260-5 *	8260-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

CONCENTRATIONS OF THE CALIBRATION STANDARDS-5.0mL OR 5.0g

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
TCL-1,8260-1	5ug/l-kg	25ug/l-kg
TCL-2,8260-2	10ug/l-kg	50ug/l-kg
TCL-3,8260-3	25ug/l-kg	125ug/l-kg
TCL-4,8260-4	50ug/l-kg	250ug/l-kg
TCL-5,8260-5	100ug/l-kg	500ug/l-kg
TCL-6,8260-6	200ug/l-kg	1000ug/l-kg

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS-25mL Purge Volume-EXAMPLES

These calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The standards are based on a volume of 25mL to achieve lower quantitation limits for the target compounds. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	25TCL -1*	25TCL -2*	25TCL-3*	25TCL -4*	25TCL-5*	25TCL-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	258260-1*	258260 -2*	258260-3*	258260-4*	258260-5*	258260-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

CONCENTRATIONS OF THE CALIBRATION STANDARDS

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
25TCL-1,25-8260-1	1.0ug/l	5.0ug/l
25TCL-2,25-8260-2	2.0ug/l	10ug/l
25TCL-3,25-8260-3	5.0ug/l	25ug/l
25TCL-4,25-8260-4	10ug/l	50ug/l
25TCL-5,25-8260-5	20ug/l	100ug/l
25TCL-6,25-8260-6	40ug/l	200ug/l

Appendix B

8260 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ storage*	Container	Hold Time
Aqueous	none; 4C	40mL no headspace	7 days
Aqueous	HCl pH<2; 4C	40mL-no headspace	14 days
Soil/solid(low level)	1g sodium bisulfite; 4C	pre-weighed 40mL vial with Teflon-lined cap	14 days
Soil/solid(high level)	none; 4C	Glass 125mL	14 days
TCLP	HCl pH<2; 4C	Tedlar bag	14 days

*storage temperature is 4C with a control criteria of less than 6C with no frozen samples

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
4-BFB 50ng on column Clock starts at injection	4-BFB 50ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification (50ug/L) RL Standard-low point on cal curve (if necessary)
Method blank	Method blank
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

4-Bromofluorbenzene Criteria

m/z	Ion Abundance Criteria
50	15.0-40.0% of m/z 95
75	30.0-60.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	Less than 2.0% of mass 174
174	Greater than 50% of mass 95
175	5.0-9.0% of mass 174
176	Greater than 95.0% but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

Appendix B

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
Less than 30% RSD	<=20% difference or drift from initial calibration

System Performance Check Compounds-SPCC

SPCC	5g/5mL minimum RRF	25mL minimum RRF
Chloromethane	0.10	0.10
1,1-Dichloroethane	0.10	0.10
Chlorobenzene	0.30	0.10
Bromoform	>0.10	>0.10
1,1,2,2-Tetrachloroethane	0.30	0.10

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

The results for all target compounds are evaluated for linearity. If the %RSD is less than 15%, the calibration is assumed linear through the origin and the average response factor can be used for quantitation. If the average response factor for the target exceeds 15% (including any CCC), the analyst must use the calibration curve option.

NOTE: The lab has the option of using a calibration curve for all analytes. This is the recommended course of action-especially for data systems that cannot automatically calculate the relative response factors.

A regression fit may be used to define the concentration/response relationship. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the curve accurately defines the concentration/response relationship over the entire calibration range.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPs for other exceptions to using non-linear curve fitting.

Recommended Internal Standards:
1,2-dichloroethane-d4; 1,4-difluorobenzene; chlorobenzene-d5; 1,4-dichlorobenzene-d4

Surrogates/System Monitoring Compounds:
dibromofluoromethane; toluene-d8; 4-bromofluorobenzene, 1,2-dichlorobenzene-d4

LCS/MS: CQAP Subset:
1,1-dichloroethene; benzene; trichloroethene; toluene; chlorobenzene

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
MS Tune Check - 50ng 4-BFB	Before initial and continuing calibration standards - every 12 hours	Mass abundances within method acceptance criteria	-Evaluate chromatogram and spectrum - Reanalyze - Retune MS and reanalyze - Remake standard and reanalyze - Perform instrument maintenance and reanalyze
Initial Calibration – minimum five point curve with lowest point at or below the Reporting Limit (RL)	Initially; after major instrument maintenance; whenever continuing calibration check fails. Prior to analysis of method blank and samples	Method criteria for CCC/SPCC (see -Calibration Acceptance Criteria – Table presented earlier in this document)	- Evaluate chromatograms, spectra, and integrations - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument maintenance and recalibrate
Continuing Calibration check - midpoint standard	Every 12 hours before analysis of method blank and samples	Method criteria for CCC/SPCC (see Calibration Acceptance Criteria - Table presented earlier in this document)	- Evaluate chromatogram, spectra, integrations - Reanalyze standard - Remake and reanalyze standard - Recalibrate - Perform instrument maintenance and recalibrate
Method Blank	Every 12 hours (per clock) before sample analyses	All reported targets <RL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Lab Control Sample (LCS) -subset of target compounds	Each batch (LCS/LCSD if MS/MSD not performed)	SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Matrix Spike/Matrix Spike Duplicate (MS/MSD) -subset of target compounds	Each batch	SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogates	All samples, blanks, LCS, MS	SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Internal Standard Area	Evaluate Continuing Calibration Check ISTD areas versus previous midpoint calibration standard; Evaluate samples, blanks, LCS, MS	-50% to +100% of area of Continuing Calibration Standard	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Reporting Limit Standard -1x to 2x the RL	(Optional) Daily. Required for Florida DEP	Detected with reasonable response	-Evaluate chromatogram, spectra, and integrations -Reanalyze -Remake standard and reanalyze -Retune and recalibrate -Perform instrument maintenance and recalibrate
Initial Demonstration of Capability	Per analyst	Method criteria	-Reanalyze targets that do not meet criteria
Method Detection Limit (MDL)	See SL SOP CA90	See SL SOP CA90	-Reanalyze and re-evaluate

Appendix C
EXAMPLE QUANTITATION REPORT

-quantitation ions
-internal standard and target compound association

COMPOUND LISTING

Method file : /chem/VM/MSI5971.i/1i0102.b/i-18260-m.m

	Compound	RT	RT Window	RF	Mass
	9 TRICHLOROTRIFLUOROETHANE(1	9.544	8.801-10.288	1.508	101.00
		9.544	8.801-10.288	0.000	151.00
		9.544	8.801-10.288	0.000	103.00
	10 1 1-DICHLOROETHENE	9.134	8.390-9.878	0.000	61.00
		9.134	8.390-9.878	0.784	96.00
		9.134	8.390-9.878	0.000	98.00
	11 IODOMETHANE	9.112	8.368-9.856	0.630	141.90
		9.112	8.368-9.856	0.000	126.90
	12 METHYLENE CHLORIDE	9.480	8.736-10.223	0.000	49.00
		9.480	8.736-10.223	0.901	84.00
		9.480	8.736-10.223	0.000	86.00
	13 CARBON DISULFIDE	9.523	8.779-10.267	2.774	76.00
		9.523	8.779-10.267	0.000	78.00
	14 ACROLEIN	8.421	7.678-9.165	0.034	56.00
		8.421	7.678-9.165	0.000	55.00
	15 trans-1 2-DICHLOROETHENE	10.408	9.664-11.152	0.000	61.00
		10.408	9.664-11.152	0.865	96.00
		10.408	9.664-11.152	0.000	98.00
	16 VINYL ACETATE	11.013	10.269-11.757	1.722	43.00
		11.013	10.269-11.757	0.000	86.00
	17 1 1-DICHLOROETHANE	10.732	9.989-11.476	1.828	63.00
		10.732	9.989-11.476	0.000	65.00
		10.732	9.989-11.476	0.000	83.00
	18 2-BUTANONE	11.424	10.680-12.167	0.383	43.00
		11.424	10.680-12.167	0.000	72.00
	19 2 2-DICHLOROPROPANE	11.770	11.026-12.513	1.387	77.00
		11.770	11.026-12.513	0.000	41.00
	20 cis-1 2-DICHLOROETHENE	11.445	10.701-12.189	0.000	61.00
		11.445	10.701-12.189	0.926	96.00
		11.445	10.701-12.189	0.000	98.00
M	21 1,2-DICHLOROETHENE (total)	7.590	7.016-8.164	0.896	100.00
	22 ACRYLONITRILE	9.501	8.757-10.245	0.247	53.00
		9.501	8.757-10.245	0.000	52.00
		9.501	8.757-10.245	0.000	51.00
	23 CHLOROFORM	11.726	10.982-12.470	1.669	83.00
		11.726	10.982-12.470	0.000	85.00
		11.726	10.982-12.470	0.000	47.00
	24 BROMOCHLOROMETHANE	11.640	10.896-12.383	0.000	49.00
		11.640	10.896-12.383	0.000	129.90
		11.640	10.896-12.383	0.418	127.90

COMPOUND LISTING

Method file : /chem/VM/MSI5971.i/1i0102.b/i-18260-m.m

	Compound	RT	RT Window	RF	Mass
	93 CHLOROPRENE	11.010	10.278-11.742	0.000	53.00
		11.010	10.278-11.742	0.000	88.00
$	25 DIBROMFLUOROMETHANE	11.878	11.134-12.621	0.000	110.90
		11.878	11.134-12.621	0.000	81.00
		11.878	11.134-12.621	1.127	112.90
*	26 1,4-DIFLUOROBENZENE	13.477	12.668-14.285	0.000	114.00
		13.477	12.668-14.285	0.000	63.00
		13.477	12.668-14.285	0.000	88.00
	27 1 1 1-TRICHLOROETHANE	12.591	11.782-13.399	0.576	97.00
		12.591	11.782-13.399	0.000	99.00
		12.591	11.782-13.399	0.000	61.00
	28 1 1-DICHLOROPROPENE	12.829	12.020-13.637	0.525	75.00
		12.829	12.020-13.637	0.000	110.00
		12.829	12.020-13.637	0.000	77.00
	29 CARBON TETRACHLORIDE	13.045	12.236-13.853	0.461	116.90
		13.045	12.236-13.853	0.000	118.90
		13.045	12.236-13.853	0.000	120.90
	30 1 2-DICHLOROETHANE	12.504	11.696-13.313	0.500	62.00
		12.504	11.696-13.313	0.000	49.00
		12.504	11.696-13.313	0.000	64.00
	31 BENZENE	13.131	12.322-13.940	1.275	78.00
		13.131	12.322-13.940	0.000	50.00
		13.131	12.322-13.940	0.000	51.00
	32 TRICHLOROETHENE	13.995	13.187-14.804	0.352	129.90
		13.995	13.187-14.804	0.000	95.00
		13.995	13.187-14.804	0.000	131.90
	33 1 2-DICHLOROPROPANE	13.952	13.144-14.761	0.520	63.00
		13.952	13.144-14.761	0.000	76.00
		13.952	13.144-14.761	0.000	65.00
	34 BROMODICHLOROMETHANE	14.082	13.273-14.890	0.602	83.00
		14.082	13.273-14.890	0.000	85.00
		14.082	13.273-14.890	0.000	129.00
	35 DIBROMOMETHANE	13.887	13.079-14.696	0.337	93.00
		13.887	13.079-14.696	0.000	173.80
		13.887	13.079-14.696	0.000	95.00
	36 2-CHLOROETHYL VINYL ETHER	14.687	13.878-15.495	0.334	63.00
		14.687	13.878-15.495	0.000	65.00
		14.687	13.878-15.495	0.000	106.00

COMPOUND LISTING

Method file : /chem/VM/MSI5971.i/1i0102.b/i-18260-m.m

	Compound	RT	RT Window	RF	Mass
	37 4-METHYL-2-PENTANONE (MIBK	15.205	14.397-16.014	0.528	43.00
		15.205	14.397-16.014	0.000	57.00
		15.205	14.397-16.014	0.000	58.00
	38 cis-1,3-DICHLOROPROPENE	14.989	14.181-15.798	0.662	75.00
		14.989	14.181-15.798	0.000	77.00
		14.989	14.181-15.798	0.000	110.00
$	39 TOLUENE-d8	16.005	15.196-16.813	1.095	98.00
		16.005	15.196-16.813	0.000	100.00
		16.005	15.196-16.813	0.000	70.00
	40 TOLUENE	16.113	15.304-16.922	0.000	91.00
		16.113	15.304-16.922	0.730	92.00
		16.113	15.304-16.922	0.000	65.00
	41 trans-1,3-DICHLOROPROPENE	15.616	14.807-16.424	0.478	75.00
		15.616	14.807-16.424	0.000	77.00
		15.616	14.807-16.424	0.000	110.00
	42 1 1 2-TRICHLOROETHANE	15.854	15.045-16.662	0.000	97.00
		15.854	15.045-16.662	0.296	83.00
		15.854	15.045-16.662	0.000	99.00
	43 1 3-DICHLOROPROPANE	16.200	15.391-17.008	0.675	76.00
		16.200	15.391-17.008	0.000	78.00
		16.200	15.391-17.008	0.000	41.00
	44 1 2-DIBROMOETHANE	16.956	16.147-17.765	0.447	107.00
		16.956	16.147-17.765	0.000	109.00
*	45 CHLOROBENZENE-d5	18.209	17.116-19.301	0.000	117.00
		18.209	17.116-19.301	0.000	82.00
		18.209	17.116-19.301	0.000	119.00
	46 2-HEXANONE	16.502	15.409-17.595	0.658	43.00
		16.502	15.409-17.595	0.000	58.00
	47 TETRACHLOROETHENE	17.215	16.123-18.308	0.000	165.90
		17.215	16.123-18.308	0.701	163.90
		17.215	16.123-18.308	0.000	167.90
	48 DIBROMOCHLOROMETHANE	16.610	15.518-17.703	0.637	128.90
		16.610	15.518-17.703	0.000	126.90
		16.610	15.518-17.703	0.000	130.90
	49 CHLOROBENZENE	18.274	17.181-19.366	1.301	112.00
		18.274	17.181-19.366	0.000	77.00
		18.274	17.181-19.366	0.000	51.00
	50 1 1 1 2-TETRACHLOROETHANE	18.111	17.020-19.202	0.000	130.90
		18.111	17.020-19.202	0.000	132.90
		18.111	17.020-19.202	0.000	119.00

COMPOUND LISTING

Method file : /chem/VM/MSI5971.i/1i0102.b/i-18260-m.m

	Compound	RT	RT Window	RF	Mass
	51 ETHYL BENZENE	18.576	17.483-19.669	2.279	91.00
		18.576	17.483-19.669	0.000	106.00
		18.576	17.483-19.669	0.000	51.00
	52 m,p-XYLENE	18.900	17.807-19.992	0.000	91.00
		18.900	17.807-19.992	0.829	106.00
		18.900	17.807-19.992	0.000	77.00
	53 o-XYLENE	19.527	18.434-20.619	0.000	91.00
		19.527	18.434-20.619	0.844	106.00
		19.527	18.434-20.619	0.000	77.00
M	54 XYLENE (total)	15.657	15.083-16.231	0.844	106.00
	55 STYRENE	19.419	18.326-20.511	1.456	104.00
		19.419	18.326-20.511	0.000	78.00
		19.419	18.326-20.511	0.000	103.00
	56 BROMOFORM	19.073	17.980-20.165	0.822	172.80
		19.073	17.980-20.165	0.000	170.80
		19.073	17.980-20.165	0.000	173.80
	57 1 1 2 2-TETRACHLOROETHANE	19.527	18.434-20.619	1.337	83.00
		19.527	18.434-20.619	0.000	85.00
		19.527	18.434-20.619	0.000	168.00
	94 PENTACHLOROETHANE	21.072	19.996-22.146	0.000	167.00
		21.072	19.996-22.146	0.000	130.00
		21.072	19.996-22.146	0.000	130.00
*	58 1,4-DICHLOROBENZENE d4	22.230	20.896-23.564	0.000	150.00
		22.230	20.896-23.564	0.000	152.00
		22.230	20.896-23.564	0.000	115.00
	59 trans-1,4-DICHLORO-2-BUTEN	19.787	18.452-21.120	0.349	53.00
		19.787	18.452-21.120	0.000	88.00
		19.787	18.452-21.120	0.000	89.00
	60 ISOPROPYLBENZENE	20.089	18.755-21.423	2.785	105.00
		20.089	18.755-21.423	0.000	120.00
		20.089	18.755-21.423	0.000	77.00
$	61 p-BROMOFLUOROBENZENE	20.154	18.820-21.488	1.365	95.00
		20.154	18.820-21.488	0.000	174.00
		20.154	18.820-21.488	0.000	176.00
	62 1 2 3-TRICHLOROPROPANE	19.743	18.409-21.077	0.341	110.00
		19.743	18.409-21.077	0.000	112.00
	63 n-PROPYLBENZENE	20.803	19.469-22.136	3.183	91.00
		20.803	19.469-22.136	0.000	120.00
		20.803	19.469-22.136	0.000	65.00

COMPOUND LISTING

Method file : /chem/VM/MSI5971.i/1i0102.b/i-18260-m.m

Compound	RT.	RT Window	RF	Mass
64 BROMOBENZENE	20.479	19.145-21.812	0.000	77.00
	20.479	19.145-21.812	0.850	156.00
	20.479	19.145-21.812	0.000	158.00
65 2-CHLOROTOLUENE	20.933	19.598-22.266	2.217	91.00
	20.933	19.598-22.266	0.000	126.00
	20.933	19.598-22.266	0.000	63.00
66 1 3 5-TRIMETHYLBENZENE	21.279	19.945-22.612	2.210	105.00
	21.279	19.945-22.612	0.000	120.00
	21.279	19.945-22.612	0.000	77.00
67 4-CHLOROTOLUENE	21.062	19.728-22.396	2.928	91.00
	21.062	19.728-22.396	0.000	126.00
	21.062	19.728-22.396	0.000	63.00
68 tert-BUTYLBENZENE	21.711	20.377-23.044	1.661	119.00
	21.711	20.377-23.044	0.000	91.00
	21.711	20.377-23.044	0.000	134.00
69 1 2 4-TRIMETHYLBENZENE	21.884	20.550-23.217	2.028	105.00
	21.884	20.550-23.217	0.000	120.00
	21.884	20.550-23.217	0.000	77.00
70 sec-BUTYLBENZENE	22.057	20.723-23.390	3.406	105.00
	22.057	20.723-23.390	0.000	134.00
	22.057	20.723-23.390	0.000	91.00
71 p-ISOPROPYLTOLUENE	22.360	21.026-23.694	2.391	119.00
	22.360	21.026-23.694	0.000	134.00
	22.360	21.026-23.694	0.000	91.00
72 1 3-DICHLOROBENZENE	22.165	20.831-23.499	1.112	146.00
	22.165	20.831-23.499	0.000	148.00
	22.165	20.831-23.499	0.000	111.00
73 1 4-DICHLOROBENZENE	22.273	20.939-23.607	1.792	146.00
	22.273	20.939-23.607	0.000	148.00
	22.273	20.939-23.607	0.000	111.00
74 n-BUTYLBENZENE	23.009	21.675-24.343	2.973	91.00
	23.009	21.675-24.343	0.000	92.00
	23.009	21.675-24.343	0.000	134.00
75 1 2-DICHLOROBENZENE	22.836	21.502-24.169	1.430	146.00
	22.836	21.502-24.169	0.000	148.00
	22.836	21.502-24.169	0.000	111.00
76 1 2-DIBROMO-3-CHLOROPROPAN	23.593	22.259-24.926	0.461	75.00
	23.593	22.259-24.926	0.000	156.90
	23.593	22.259-24.926	0.000	154.90

COMPOUND LISTING

Method file : /chem/VM/MSI5971.i/1i0102.b/i-18260-m.m

Compound	RT	RT Window	RF	Mass
77 1 2 4-TRICHLOROBENZENE	25.883	24.549-27.217	1.606	179.90
	25.883	24.549-27.217	0.000	181.90
	25.883	24.549-27.217	0.000	145.00
78 HEXACHLOROBUTADIENE	26.467	25.133-27.801	1.332	224.80
	26.467	25.133-27.801	0.000	222.80
	26.467	25.133-27.801	0.000	189.90
79 NAPHTHALENE	26.359	25.025-27.692	2.172	128.00
	26.359	25.025-27.692	0.000	102.00
	26.359	25.025-27.692	0.000	51.00
80 1 2 3-TRICHLOROBENZENE	26.727	25.634-27.819	1.369	180.00
	26.727	25.634-27.819	0.000	182.00
	26.727	25.634-27.819	0.000	109.00
86 ACETONITRILE	8.573	7.829-9.316	0.079	41.00
	8.573	7.829-9.316	0.000	40.00
87 3-CHLORO-1-PROPENE	9.588	8.844-10.331	1.070	41.00
	9.588	8.844-10.331	0.000	76.00
88 PROPIONITRILE	10.948	10.205-11.692	0.081	54.00
	10.948	10.205-11.692	0.000	55.00
89 METHACRYLONITRILE	11.488	10.745-12.232	0.305	67.00
	11.488	10.745-12.232	0.000	52.00
90 ISOBUTANOL	12.180	11.436-12.924	0.046	43.00
	12.180	11.436-12.924	0.000	41.00
91 METHYL METHACRYLATE	14.341	13.533-15.150	0.201	69.00
	14.341	13.533-15.150	0.000	41.00
92 ETHYL METHACRYLATE	16.286	15.193-17.378	0.619	69.00
	16.286	15.193-17.378	0.000	41.00
	16.286	15.193-17.378	0.000	39.00
95 METHYL T-BUTYL ETHER	11.250	10.663-12.155	0.000	73.00
	11.250	10.663-12.155	0.000	57.00
96 TETRAHYDROFURAN	1.000	0.500-1.500	0.000	42.00
	1.000	0.500-1.500	0.000	72.00
	1.000	0.500-1.500	0.000	0.00
97 1 4-DIOXANE	1.000	0.980-1.020	0.000	88.00
	1.000	0.980-1.020	0.000	58.00
98 2-PICOLINE	1.000	0.980-1.020	0.000	93.00
	1.000	0.980-1.020	0.000	66.00

Approval Signature: R. Wayne Robbins	
Title : Corporate QA Manager	Date : 1/14/99

CHLORINATED HERBICIDES
(615 and 8151A)

1.0 SCOPE AND APPLICATION

1.1 This procedure can be used to determine the concentration of various chlorinated herbicides in water, groundwater, leachate, soil, sediment, and waste samples extracts. Table 1 lists the target compounds and the retention times for each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria are given in Section 5 of the current revisions of the Savannah Laboratories' quality assurance plans.

2.0 SUMMARY OF METHOD

2.1 A known volume or weight of sample is extracted in accordance with SL SOP EX45. The extracted chlorinated herbicide methyl derivatives are analyzed by gas chromatography (GC) configured with dual capillary columns and dual electron capture (EC) detectors. This configuration allows for simultaneous detection and confirmation of the herbicides. Identification of the target compounds in samples is done by comparing the retention times of the peaks with standards analyzed under the same GC conditions.

GC/MS confirmation may be employed if the target compound concentration is sufficiently high or if the extract is concentrated to an appropriate final volume. The esterified extract must be used for the GC/MS confirmation-do not use the 8270 extract.

2.2 This SOP is based on the guidance in SW-846 Method 8000B and Method 8151A and EPA Method 615.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and eye protection(lab glasses or face shields), must be worn. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

3.4 Diethyl ether is a flammable solvent and it must be used in a well-ventilated hood or extraction area. The solvent vapors will tend to accumulate along the floor. High concentrations of diethyl ether can cause drowsiness, dizziness, and headache.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. The glassware must be scrupulously cleaned (SL SOP AN60: Glassware Cleaning Procedures). All of the materials and reagents must be demonstrated to be free from contaminants by the analysis of reagent blanks (method blanks). Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences. The extraction procedure described in SL SOP EX45 has several steps that are designed to eliminate or minimize interferences due to sample matrix.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Aqueous samples are routinely collected in 1-L amber glass containers equipped with Teflon-lined caps. No preservative is required. The sample is iced at the time of collection and stored at 4C (less than 6C with no frozen samples) in the lab. Sample must be extracted within seven (7) days of collection and the extract must be analyzed within forty (40) days of extraction.

TCLP leachate samples are stored in the same manner as aqueous field samples. The extraction must be performed within seven(7) days of the leaching procedure and the extract analyzed within forty(40) days of extraction

5.2 Soils, sediments, sludges, and wastes are collected in glass containers equipped with Teflon-lined caps. The routine container is 500-mL glass. Larger or smaller containers may be supplied. No preservative is required. The samples are iced at the time of collection and stored at 4C (less than 6C with no frozen samples) in the lab. The hold time for herbicides in solid and non-aqueous matrices is 14 days from the date of collection. The extract must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph equipped with dual electron capture detectors and automatic liquid samplers.

6.2 Recommended Columns:

J&W DB-5 fused silica column, 30M x 0.53mm ID, 1.5um film
J&W DB-608 fused silica column, 30M x 0.53mm ID, 0.83um film

6.3 Data system compatible with the GC and capable of detecting and storing chromatographic data. Nelson 2600 or equivalent

6.4 Autosampler vials, septa, and caps

6.5 Microsyringes- 10-uL, 25-uL, 50-uL, 100-uL

7.0 REAGENTS

Hexane: Pesticide grade or equivalent

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all neat compounds or stock solutions. All preparation steps must be in accordance with SL SOP AN41:*Standard Material Traceability*.

Preparation of the Calibration Standards

If the stock standard is prepared using the free acid form, no correction is required for the concentration. The concentration of the standard is based on the weight of free acids added per unit volume prior to derivitization. A stock standard prepared from neat herbicide acids has to be derivitivized prior to analysis. If a herbicide standard is purchased as a neat methyl ester, the concentration of the standard must be corrected to the free acid concentration. See SL SOP AN43: Standard Preparation for guidance on the preparation of standards.

The calibration standards must be corrected to the weight of the acid. This will eliminate the need to correct the final concentration of the sample. The correction factors are given in Table 2.

The stock standards are prepared in hexane from either the stock standard mixes purchased from vendors or from the individual stock standards prepared in-house. An example of the preparation of the calibration standard is given in Table 3

9.0 SAMPLE PREPARATION

Sample preparation steps are given in SL SOP EX45.

10.0 PROCEDURE

10.1 GC Conditions

The columns and analytical conditions listed in this section are given for guidance. The lab must document the actual columns and conditions used for each analysis in the instrument maintenance, data system, or on the sample log.

10.1.1 Recommended Columns:

J&W DB-5 fused silica column, 30M x 0.53mm ID, 1.5um film
J&W DB-608 fused silica column, 30M x 0.53mm ID, 0.83um film

The columns are connected to a single injection port with either a glass Tee-splitter or a glass y-splitter. This configuration allows for the simultaneous analysis and confirmation of the target compounds.

Carrier gas flow: He at approximately 7mL/min (per column)
Make-up gas flow: N2 at approximately 25mL/min (per detector)

10.1.2 Suggested GC temperature programs

APP 9 temperature program:
Initial temperature: 170 C
Initial hold time: 4 minutes
Program rate: 8 C / minute
Final temperature: 280 C
Final hold: 2 minutes
TOTAL TIME: 20 minutes

8151 Full List temperature program
Initial temperature: 100 C
Initial hold time: 2 minutes
Program rate: 8 C / minute
Final temperature : 280 C
Final hold: 2 minutes
TOTAL TIME: 27 minutes

10.1.3 Heated zone temperatures

Injector: 240-260 C
Detector: 300C

10.1.4 Injection volume: 2-4uL (1-2-uL per column)

10.2 Initial Calibration

The external standard calibration technique is routinely employed for the determination of the concentration of herbicides. The lab also has the option of using internal standard calibration. Pentachloronitrobenzene (PCNB) may be a suitable compound to use as an internal standard

10.2.1 Prepare and inject the calibration standards using the guidelines listed in Section 8 of this SOP. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in Section 10.3.

Note that the following offers two (2) options for calibration and quantitation – average RF or regression curve. Only one need be chosen per analyte.

When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

10.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed?

>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?

>Are the peaks symmetrical? Is there tailing or fronting?

>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.3 Average RF/CF Option:

Calculate the response factor of each calibration standard, the average response factor and the relative standard deviation using the following equations:

Response Factor-external standard

$$X_{RF} = \frac{mg/mL}{area} = \frac{mg}{mL\ area}$$

Note that the inverse relationship can also be used to evaluate the response of the detector. Dividing the area of the standard by the concentration is referred to as the calibration factor.

Response factor-internal standard

$$RF = \frac{Ac}{Ais} \otimes \frac{Cis}{Cc}$$

where

Ac = area of the target compound
Ais = area of the internal standard
Cc = concentration or mass on-column of the target compound (ug/mL)
Cis = concentration or mass on-column of the internal standard (ug/mL)

Average Response Factor

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 + ..RFn}{n}$$

Relative Standard Deviation

$$\%RSD = \frac{standard\ deviation}{\overline{RF}} \otimes 100$$

and

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}}$$

Where

RF_i = response factor of the individual calibration level
$\overline{RF}$ = average response factor

Initial Calibration Criteria:

600-series: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear and the average response factor (or calibration factor) may be used for quantitation.

8000-series:If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear and the average response factor (or calibration factor) may be used for quantitation.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

10.2.4 Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPs for other exceptions to using non-linear curve fitting.

10.3 Calibration Verification

Calibration is verified every 24 hours for 600-series methods and every 12 hours or 20 samples, whichever is more frequent, for 8000-series methods by the analysis of continuing calibration check standards. Note that the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive for the compound(s) failing high are detected, these samples may be reported.

10.3.1 Analyze the mid-level standard(s). Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration:

$$\%Drift = \frac{result - expected}{expected} \times 100$$

Where
result = concentration or nanograms on-column of the calibration check standard quanted against the curve
expected = true concentration or nanograms on-column of the calibration check standard

$$\%Difference = \frac{RF_{init} - RF_{cont}}{RF_{init}} \times 100$$

Where
RF_{init} = average response factor from the initial calibration curve
Rf_{cont}= response factor from the continuing calibration standard

---

10.3.2 Continuing Calibration Verification Criteria

If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

600-series: If the percent drift or percent difference is less than or equal to 10%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

8000-series: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor is used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

10.4 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the SOP Summary after Section 15.

10.4.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks (hexane) may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.4.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. For the single point multicomponent products, the extract should be diluted until the area is no more than a factor of two above the area of the single point standard (see section 11.1 for quantitation of multi-peak target compounds).

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used (*F34 or *F42) or qualification in a case narrative provided to the client. For TCLP analyses, every effort should be made to achieve the regulatory level without substantial instrument overload.

For clients who demand we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve (i.e., a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client). Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SL SOP AN66: Determination of Retention Time Windows for Gas Chromatographic Analyses. If internal standard calibration is used, the determination of absolute retention time windows is not required. Relative retention times, as described in Section 11.4, are used to identify the target compounds.

11.0 DATA ANALYSIS/CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times), the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration, and the integration of the peaks. Manual integration must be documented in accordance with SL SOP AN65. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data.

The judgement and experience of the analyst and his/her colleagues is an important part of the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks correctly integrated?
- Are there co-eluting peaks or matrix interferences?

11.1 Qualitative analysis

Identification of the target compounds is based on retention time. The analyst should scan the sample chromatogram for the target compounds on the primary analytical column. The analyst should use the retention time (RT) window calculated around the CCV as guidance for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds to help gauge possible shifts in the RT of the target compounds. See SL SOP AN66.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peaks areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (10.3.2).

11.1.1 The surrogate should be evaluated first to check for shifts in retention times and to evaluate the surrogate recovery.

The extract contains the surrogate Dichloroacetic acid (DCAA). The recovery criteria are given in Section 5 of the SL QAPs.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Generally, it is expedient to calculate the recovery on one chromatogram. If this recovery is acceptable, the other channel is not evaluated for recovery. If interferences are suspected, evaluate recovery on the other channel. Note that given this nature, an extract is considered acceptable if one of the 2 potentially calculated recoveries is acceptable.

NOTE: If the recovery of the surrogate is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to section 13 of the current QA Plan regarding this issue.

11.1.2 Label and calculate the concentration of each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences and the overall shapes of the peaks.

If the concentration is below the detection limit, the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.3.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds to help gauge possible shifts in the RT of the target compounds.

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

---

If the relative percent difference is less than 40%, the presence of the target compound is confirmed and the lower concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A large relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. If interference is detected, flag the result to note the disparity between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

The following table summarizes the general guidance for the evaluating of chromatographic data. The table assumes that the calibration criteria have been met and that the sample has acceptable associated surrogate and lab spike recoveries.

PEAK INFORMATION	ACTION	REPORT*
No peaks found on primary or secondary column		Report < RL
Peak found within RTW on primary column	Peak is tentatively identified as the target	If concentration < RL, report < RL If concentration > RL, evaluate confirmation column.
Peak found within RTW on confirmation column	Peak is confirmed as the target	If concentration < RL, report <RL If concentration >RL, calculate %RPD -if %RPD <40%, report lower concentration of primary and confirmation analyses. -if %RPD >40%, flag result to note the disparity Case narrative or note to PM may be required for complex matrices.

*RL may be the SL Reporting Limit in Table 5 of the CQAP or may be defined by the client QAP or contract.

The analyst must clearly show how the reported sample results were determined.

11.1.4 Identification "Tools"

Analysis by GC/MS (scan or SIM) may be used to confirm the presence of chlorinated herbicides (see SL SOP SM06: *Guidelines for SIM Analysis by GC/MS.)* The herbicide extract must be used to perform the GC/MS confirmation-unesterified extracts (e.g., the GC/MS extract) are not suitable for herbicide analysis or confirmation.

11.1.4.1 Relative Retention Time

The retention time of a surrogate compound provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions.. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification.

11.1.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the sinitial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience. The extract must be fortified with herbicides as methyl esters-unesterified herbicides are not amenable to GC analysis.

11.2 Calculations-External Standard

Aqueous/Liquid Samples

If the regression curve option is used, the sample concentration is calculated:

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{V}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

If the response factor option is chosen for quantitation:

$$Concentration(ug/L) = \frac{ug/mL}{response} \otimes response \otimes \frac{F \otimes DF}{V}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

Soils/Solids Samples

Regression Curve

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
W = weight of sample extracted (kg)
solids = (percent solids)/100

If the response factor option is chosen for quantitation:

$$Concentration(ug/kg, dw) = \frac{ug/mL}{response} \otimes response \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)
W = weight of sample extracted (kg)
solids = (percent solids)/100

11.3 Internal Standard Calibration

Aqueous Samples

$$Concentration(ug/L) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{V} \otimes DF$$

where
Ac = area of the target compounds
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
V = volume of sample extracted (l)
DF =dilution factor

Soil Samples

$$Concentration(ug/kg, dw) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{W \otimes solids} \otimes DF$$

where
Ac = area of the target compounds
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
W =weight of sample extracted (kg)
Solids = (percent solids)/100
DF =dilution factor

12. 0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The **analytical batch** is discussed in SL SOP AN02: *Analytical Batching*, and these criteria are summarized in the SOP Summary included in this SOP. Calculation of QC is also given in AN02.

12.2 The **method detection limit (MDL)** is defined as the concentration of the an analyte that can be measured with a 99% confidence that the result is greater than zero. See SL SOP CA90: *Procedure for Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items are included in this revision. See Section 10 of the current SL QAP.

14.0 TROUBLESHOOTING

No items are included in this revision.

15.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

2. *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office: Washington, DC, July 1, 1988.

3. Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*, current revision.

METHOD SUMMARY

HOLD/STORAGE

Parameter	Aqueous	Soils/Solids/Wastes
Routine Container	1-L amber glass fitted with Teflon-lined cap	500-mL or smaller glass fitted with Teflon-lined cap
Preservative	None	None
Hold time	7 days from date of collection; 40 days from date of extraction	14 days from date of collection; 40 days from date of extraction
Storage	4C (less than 6C with no frozen samples) from collection to analysis	4C (less than 6C with no frozen samples) from collection to analysis

EXTRACTION

Aquoues-500mL of sample; separatory funnel extraction at pH >12 with diethyl ether followed extraction at pH<2 with diethyl ether; esterify and dilute to 10mL final volume with hexane

Soils- acidify 30g of sample mixed with acidified sodium sulfate; sonication or Soxhlet extraction with 1:1methylene chloride/acetone; concentrate and hydrolyze with KOH, treat same as aqueous sample from this point forward.

SEQUENCE

615

Initial Calibration Standards
Client samples analyzed until 24 hour clock expires
Calibration Verification standard – mid-level concentration
Client samples analyzed until 24 hour clock expires

8151

Initial Calibration Standards
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration

The sequence continues until all samples are analyzed or until the CCV standard fails to meet the acceptance criteria.

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration- 5 point minimum with lowest point at RL	Prior to sample analysis or when CCV fails	1) RSD of each target <= 20%(8151) or 10% RSD (615); OR 2) plot regression curve (CC>=0.99) (see Section 10.2 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification(CCV)	After every twenty sample analyses (or 12 hours) and at the end of the sequence	8151: Percent difference or drift <= 15% 615: Percent difference or drift <= 10% (see Section 10.3 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in Table 5 of the SL CQAP	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Lab control sample (LCS)- Subset of targets in SL CQAP	Per batch	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix spike(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogate	All samples, method blanks, and QC	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Reporting limit(RL) standard-lowest level calibration standard	Daily-required for Fla DEP	Detected with reasonable sensitivity	-Reanalyze RL standard -Remake and reanalyze RL standard -Perform instrument or column maintenance, recalibrate, and reanalyze associated samples
Initial Demonstration of Capability (IDOC)	Initially and when new analysts trained	Evaluate in accordance with method criteria	Repeat test for analytes that fail criteria
Method Detection Limit (MDL)	See SL SOP CA90	Evaluate in accordance with SL SOP CA90	Evaluate in accordance with SL SOP CA90
Retention time window determination	See SOP AN66	See SOP AN66	See SOP AN66

TABLE 1-"full list"		
Compound	RT COL 1	RT COL 2
Dalapon	2.90	3.35
DCAA (surrogate)	13.18	13.41
MCPP	13.56	14.08
Dicamba	13.66	13.63
MCPA	14.30	14.44
Dichlorprop	14.94	15.36
2,4-D	16.10	15.89
2,4,5-TP (Silvex)	18.27	19.22
2,4,5-T	20.49	20.33
Dinoseb	21.00	23.06
2,4-DB	22.81	23.36

COL1 = DB608 COL2 = DB-5

TABLE 1A-"short list"		
Compound	RT COL 1	RT COL 2
DCAA (surrogate)	5.38	5.75
2,4-D	7.65	7.61
2,4,5-TP (Silvex)	8.80	9.31
2,4,5-T	9.68	9.74

COL1 = DB608 COL2 = DB-5

TABLE 2

HERBICIDE MOLECULAR WEIGHTS AND CORRECTION FACTORS

Herbicide acid	MWacid	MWester/ether	Correction factor
2,4-D	221.04	235.07	0.940
Dalapon	142.97	157.00	0.911
2,4-DB	249.09	263.12	0.947
Dicamba	221.04	235.07	0.940
Dichloroprop	235.07	249.09	0.944
Dinsoeb	240.22	254.24	0.945
MCPA	200.62	214.65	0.935
MCPP	214.65	228.67	0.939
2,4,5-TP(Silvex)	269.51	283.54	0.951
2,4,5-T	255.48	269.51	0.948
DCAA	205.04	219.07	0.936
Picloram	241.48	255.51	0.945
Pentachlorophenol	266.35	280.37	0.950

Example Calculation

$$CF(2,4-D) = \frac{Wacid}{Wester} = \frac{221.04}{235.07} = 0.94$$

If the standard is expressed as mass of ester per volume, convert the concentration to he acid form by multiplying by the correction factor (CF).

Table 3-Example Standard Preparation "Recipes"

"Short List" Herbicide Intermediate Standard

STOCK STANDARD	VENDOR/ PART NO.	CONC. (ug/mL)	mL of stock	fvol (mL)	conc (ug/mL)
Herbicides	Ultra HBM-815M	100	1.0	10	10
DCAA	Ultra PPS-161	100	1.0		10

Solvent is hexane

"Short" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
"short" list herbicide intermediate std	10	50	100	200	250	500	750	1000

* microliters of intermediate standard to 10mL of hexane. Seven standards are prepared and analyzed but only 5 are used. MCPP and MCPA are difficult to calibrate at low concentrations.

"Short" list Calibration Standards

Target Compounds	1*	2*	3*	4*	5*	6*	7*
DCAA, 2,4,-D, 2,4,5-TP (Silvex), 2,4,5-T	0.05	0.10	0.20	0.25	0.50	0.75	1.0

* ug/mL

"Full" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
Ultra HBM-8150M		5	10	12.5	20	50	67	100
Dicamba, 2,4,5-TP (Silvex), 2,4,5-T	10							
2,4-D, 2,4-DB, Dichlorprop	100							
Dinoseb	50							
MCPA, MCPP	10000							

* microliters to 10mL hexane

Table 3-Example Standard Preparation "Recipes"

"Full" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
Ultra HBM-8150M								
Dicamba, 2,4,5-TP (Silvex), 2,4,5-T	10	0.005	0.010	0.0125	0.020	0.050	0.067	0.10
2,4-D, 2,4-DB, Dichlorprop	100	0.050	0.10	0.125	0.20	0.50	0.67	1.0
Dinoseb	50	0.025	0.050	0.0625	0.10	0.25	0.33	0.50
MCPA, MCPP	10000	5.0	10	12.5	20	50	67	100

*ug/mL

Approval Signature: R. Wayne Robbins	
R. Wayne Robbins	
Title: Corporate QA Manager	Date: Jan 16, 1998

PETROLEUM PRODUCTS, DRO, AND TOTAL HYDROCARBONS BY GAS CHROMATOGRAPHY MODIFIED 8015B EXTRACTABLES

1.0 SCOPE AND APPLICATION

1.1 This method may be used to determine the identification and concentration of various petroleum products in a variety of matrices. Table 1 lists the products that are routinely analyzed using this procedure: diesel, mineral spirits, kerosene, and motor oil ("heavy oil" or oil-range organics). The target compounds are generally in the carbon range of C10 to C40. Table 2 lists other products that may be determined by this method. Examples of chromatograms are included at the end of this SOP.

1.2 **It is not the intention of this SOP to replace the DRO procedures specified bv various state agencies (Tennessee, Wisconsin, Florida, or California), although the guidelines of this SOP are similar to the guidelines given in the state procedures.**

1.3 The primary scope of this SOP is identify and quantify samples based on a known or suspected hydrocarbon product. If the identification is not required, diesel range organics or total hydrocarbons may be requested. If DRO or total extractable hydrocarbons are requested, the peaks within the range of a diesel standard (C10-C28) are summed and quantified against diesel.

1.4 The method detection limit (MDL), the reporting limit, and the accuracy and precision criteria for the target compounds are listed in Section 5 of the Savannah Labs' *Comprehensive Quality Assurance Plan* and the *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 Aqueous samples are acidified and extracted by separatory funnel or continuous liquid-liquid extraction using methylene chloride. Pure products or a product layer in a water sample are diluted in methylene chloride. Soils are extracted with 1:1 acetone/methylene chloride using sonication or Soxhlet extraction. The extract is concentrated to a final volume of 1.0mL and a 2-5µL aliquot of the extract is injected onto a GC equipped with a megabore or capillary column and a flame ionization detector (FID).

2.2 The extract for the determination of petroleum products may also be used for the determination of polynuclear aromatic hydrocarbons (PAH) if the dual column/dual detector configuration is used.

2.3 This procedure is based on SW-846 Method 8015B. An SOP summary is included as a quick reference to the minimum QC required to perform this method.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or will put yourself or others in potentially hazardous situations.

3.2 Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and other equipment should be used. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

4.0 INTERFERENCES

4.1 Glassware and extracting vessels should be scrupulously cleaned and solvent rinsed to reduce artifacts that may interfere with the analysis. (see SL SOP AN60:*Glassware Cleaning*). Glassware and reagents must be demonstrated to be free from interferences by the analysis of reagent blanks.

4.2 Co-extracted compounds may interfere with the qualitative and quantitative determination of the target compounds. The major problem associated with the analysis of these types of compounds will be the presence of two or more closely eluting products in the same sample extract. The analyst must be aware that weathering takes place in environmental samples and can sometimes make identification and quantitation difficult.

4.3 Highly chlorinated water may cause loss of PAH compounds, especially when combined with the acidic extraction..

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Liquid samples are collected in amber glass containers equipped with a Teflon-lined cap. The samples are iced at the time of collection and stored in a refrigerator maintained at <6° C. No preservative is added to the sample. Liquid samples must be extracted within 7 days of sample collection and the extract must be analyzed within 40 days of extraction.

5.2 Soil and waste samples are collected in amber or clear glass containers equipped with Teflon-lined containers. The samples are iced at the time of collection and be stored in a refrigerator maintained at <6°C. Soil and waste samples must be extracted within 14 days of sampling and the extracts must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

The materials and apparatuses listed in this section are included as a general overview of the equipment used by SL to perform this analysis and to provide guidance to the lab in selecting materials to perform the analysis.

6.1 Gas Chromatographs (GC), temperatures programmable equipped with single or dual flame ionization (FID) detectors and a compatible autosampler.

6.2 Columns

DB-5 fused silica capillary 30 m x 0.53 mm ID, 1.5 um film (J&W or equivalent)

RTX-200 fused silica capillary 30 m x 0.53 mm ID 1.0 um film (J&W or equivalent)

Guard Column 6 m x 0.53 mm ID, 1.5 um film

The DB-5 column and the RTX-200 column can be connected to a single injection port for simultaneous detection and confirmation. The columns are attached to the injection port with a glass tee or y-splitter. A guard column is recommended to help protect the analytical columns if a y-splitter is used. Capillary columns (0.32mmID) may also be used but will generally not have the capacity of the megabore columns.

6.3 Microsyringes – appropriate volumes

6.4 Volumetric flasks - Class A- appropriate volumes

6.5 Storage vials, 7-mL or 12-mL, with Teflon-lined screw cap

6.6 Autosampler vials-compatible with autosampler type

7.0 REAGENTS

7.1 Methylene chloride, pesticide residue grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in SL SOP AN43:*Standard Preparation.*

8.1 The lab should purchase certified solutions from SL-approved vendors, if available. **The lab should prepare standards from neat materials only if a certified solution is not available.** See SL SOP AN43 for guidance for standard preparation.

8.2 Stock Standards

Stock standards are prepared from neat products or purchased as solutions.

Stock standards from neat products are prepared in methylene chloride by the following procedure:

$$C(mg/mL) = \frac{W}{V}$$

where

C = concentration of primary stock standard (mg/mL)
W = weight of product (mg)
V = volume of solvent used (mL)

EXAMPLE
Prepare 10mL of stock standard containing the target compound at a concentration of 100mg/mL.

$$W(mg) = C(mg/mL) \otimes V(mL) = 100mg/mL \otimes 10mL = 1000mg$$

1000mg (1.000g) of the material is transferred to a 10mL volumetric flask and diluted to volume with a suitable solvent.

The following chart lists preparation instructions for the stock solutions prepared in hexane from neat products:

Petroleum Product Neat Standard	(W_n) Weight of Neat Product (mg)	(V_p) Final Vol (mL)	(C_p) Primary Stock Standard Concentration (mg/mL)
Diesel	1000	10	100
Kerosene	1000	10	100
Mineral Spirits	1000	10	100
Heavy Oil	1000	10	100

8.3 Calibration Standards

Determine the volume of standard to be prepared and the volume of the stock standard needed to make the calibration standard. The following equation can be used:

$$Cstock \otimes Vstock = Ccal \otimes Vcal$$

$$Vstock = \frac{Ccal \otimes Vcal}{Cstock}$$

where
Vstock = volume of stock standard needed to prepare the cal standard (mL)
Cstock = concentration of stock solution(mg/mL)
Ccal = concentration of calibration standard to prepare(mg/mL)
Vcal = volume of calibration standard to prepare(mL)

The concentration can be expressed in whatever terns the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for *Cstock* and *Ccal*.

8.3.1 Products Identification and DRO

Diesel fuel is the primary calibration product and is prepared at a minimum of five levels, along with the surrogate compounds (optionally). The other petroleum products are prepared individually at a single calibration level. The calibration standard concentrations range from 0.50 mg/mL to 10 mg/mL.

The following chart gives an example of the preparation of several of the target products:

Cal Standard	StockStd Conc (mg/mL)	Vol of Stock Std (mL)	uL 2FBP and OTP* (2000ug/mL)	Final Vol of Cal Std (mL)	Conc of Cal Std (mg/mL)
Diesel –1	100	0.050	250	10	0.50
Diesel –2	100	0.100	250	10	1.0
Diesel –3	100	0.200	250	10	2.0
Diesel –4	100	0.500	250	10	5.0
Diesel –5	100	1.0	250	10	10

*surrogates are at 50ug/mL in each calibration standard

Cal Standard	StockStd Conc (mg/mL)	Vol of Stock Std (mL)	uL 2FBP and OTP* (2000ug/mL)	Final Vol of Cal Std (mL)	Conc of Cal Std (mg/mL)
Mineral spirits	100	0.10	250	10	1.0

*surrogates are at 50ug/mL

Cal Standard	StockStd Conc (mg/mL)	Vol of Stock Std (mL)	uL 2FBP and OTP* (2000ug/mL)	Final Vol of Cal Std (mL)	Conc of Cal Std (mg/mL)
Kerosene	100	0.10	250	10	1.0

*surrogates are at 50ug/mL

Cal Standard	StockStd Conc (mg/mL)	Vol of Stock Std (mL)	uL 2FBP and OTP* (2000ug/mL)	Final Vol of Cal Std (mL)	Conc of Cal Std (mg/mL)
Heavy oil	100	0.50	250	10	5.0

*surrogates are at 50ug/mL

8.3.2 A straight chain alkane calibration standard which contains the even carbon alkanes from C8- C28 is prepared. Each alkane in the standard solution is prepared at a concentration of 100 ug/mL

The alkane standard is used to aid the analyst in identifying the target products and to provide additional information about the sample when positive product identification is not possible.

8.3.3 The surrogates, 2-fluorobiphenyl and o-terphenyl, can be calibrated separately or included in the product working level standards at the discretion of the lab. It is recommended that the surrogates be calibrated separately to avoid identification and integration errors that may occur if the surrogates are included in a product(s) that has peaks that coelute with the surrogate. The following table gives an example preparation of the surrogate calibration standard:

surrogate cal std	uL of 2-fluorobiphenyl (2000ug/mL)	uL of o-terphenyl (2000ug/mL)	final vol in MeCl2 (mL)	conc. of surrogates (ug/mL)
DROSUR	25	25	1.0	50

Larger volumes of the calibration standards may be prepared at the discretion of the lab.

9.0 SAMPLE PREPARATION

The sample preparation procedures are described in the following SL SOPs:

Procedure	Matrix	SL SOP
Continuous Liquid-liquid extraction	aqueous	EX30
Separatory funnel extraction	aqueous	EX35
Ultrasonic extraction	soils and sediments	EX40
Waste dilution	waste samples (oils, products, etc)	EX42
Zymark extract concentration	aqueous, leachate, and soil extracts	EX50

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions

These conditions and parameters are given for guidance. The conditions and parameters may be modified to optimize the analytical system.

10.1.1 Two configurations may be used for the analysis of the DRO/petroleum products. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column or a glass tee.

DB-5 fused silica capillary 30 m x 0.53 mm ID, 1.5 um film (J&W or equivalent)

RTX-200 fused silica capillary 30 m x 0.53 mm ID 1.0 um film (J&W or equivalent)

Guard Column 6 m x 0.53 mm ID, 1.5 um film

A guard column is recommended to help protect the analytical columns. Capillary columns (0.32mmID) can also be used but will generally not have the capacity of the megabore columns.

10.1.2 GC Parameters

Injector: 240- 280° C
Detector: FID at 300° C
Carrier Gas Flow: Helium at 5 mL/min (per column)
Make-up Gas Flow: Helium or nitrogen at 25 mL/min (per detector)

NOTE: Adjust the air, hydrogen, and carrier gas/makeup gas flows to the optimum rates suggested by the GC manufacturer.

Temperature program (example chromatogram temperature program):
Initial Temp: 90° C
Initial Hold: 4.0 min
Program Rate: 10° C/min
Final Temp: 290° C (hold for 10 minutes)
Injected Volume: 4.0 uL - 2.0 uL per column (single injection into guard column and "Y" splitter
Run Time: Approximately 40 min.

10.2 Calibration
The external standard calibration technique is employed for the determination of the concentration of petroleum products. If the PAH compounds are also to be determined using the same extract, refer to SL SOP SG40.

10.2.1 The **initial calibration** for identification and quantitation of products will involve the analysis of a five-point calibration for either diesel or the site-specific product and single point calibration standards for the carbon range standard and the other products to be determined for the set of samples being analyzed.

10.2.1.1 Prepare the calibration standards using the guidelines listed in Section 8 of this SOP and analyze the standards using the instrument conditions that are optimized for the analysis. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards.

10.2.1.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the methylene chloride blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?

>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?

>Are the peaks symmetrical? Is there tailing or fronting?

>Are the areas of the peaks normal for the sensitivity setting being used?

10.2.1.3 Tabulate the total area for each of the initial calibration standard(s) (either the diesel or the site-specific fuel or the single point product standards). **Quantitation of sample concentration for the multi-component products is based on the total area (including baseline integration) of all the peaks in the retention time range defined by the reference petroleum standard.** Note that the quantitation of the diesel or site-specific product will be based on the calibration curve while the quantitation of the other products will be based on a single point calibration standard.

10.2.1.4 Calculate the response factor for each calibration standard using the following equation:

$$X_{RF} = \frac{mg/mL}{area} = \frac{mg}{mL\ area}$$

Note that the inverse relationship can also be used to evaluate the response of the detector. Dividing the area of the standard by the concentration is referred to as the calibration factor.

10.2.1.5 Calculate the average response (or calibration) factor for the five point diesel or site specific product.

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 + RF_4 + RF_5}{5}$$

10.2.1.6 Calculate the standard deviation of the five calibration levels for each target.

$$Standard\ Deviation = \sqrt{\left|\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}\right|}$$

where
RF_i = response factor of the individual calibration level
$\overline{RF}$ = average response factor

10.2.1.7 Calculate the relative standard deviation (% RSD) of the five levels for each target:

$$\%\ RSD = \frac{standard\ deviation}{\overline{RF}} \otimes 100$$

If the % RSD is less than 20% for the diesel, site-specific product, or TPH in the five-point initial curve, the calibration is considered linear and the average response factor (or calibration factor) is used for quantitation.

10.2.1.8 Alternatively, a calibration curve can be established for each analyte by plotting the concentration (or nanograms on-column) along the x-axis and the corresponding response along the y-axis. A linear or higher order calibration curve can be evaluated and a curve with a 0.99 or higher correlation coefficient can be used for quantitation.

10.2.2 The continuing calibration check standard is either the mid-level diesel standard or the mid level site-specific product standard.

10.2.2.1 Analyze the mid level diesel or site-specific product. Tabulate the total area of the target analyte and calculate the response factor:

$$X_{RF} = \frac{mg/mL}{area} = \frac{mg}{mL\ area}$$

Note that the inverse relationship can also be used to evaluate the response of the detector. Dividing the area of the standard by the concentration is referred to as the calibration factor.

Calculate the percent difference(%D) of between the initial and continuing calibration response factors:

$$\%Difference = \frac{RF_{init} - RF_{cont}}{RF_{init}} x\ 100$$

where

RF_{init} = average response factor from the initial calibration curve

Rf_{cont}= response factor from the continuing calibration standard

If the percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor is used for quantitation.

If the percent difference is greater than 15%, another continuing calibration standard should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the continuing calibration criteria is not met, a new initial calibration must be prepared and analyzed.

10.2.3 If the calibration curve option has been chosen, calculate the percent drift (%D) of between the initial and continuing calibration response based on the true or theoretical concentration of the check standard:

$$\%Drift = \frac{result - expected}{expected} x\ 100$$

where

result = concentration or nanograms on-column of the calibration check standard quanted against the curve

expected = true concentration or nanograms on-column of the calibration check standard

If the percent drift is less than or equal to 15%, the calibration curve is verified and can be used for quantitation.

If the percent difference is greater than 15%, another continuing calibration standard should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the continuing calibration criteria is not met, a new initial calibration must be prepared and analyzed.

10.3 Sample Analysis Sequence

The following sequence defines the order of events that should be followed for this analysis.

STANDARD/SAMPLES
Initial Calibration-diesel or site specific product at 5 levels; mineral spirits, kerosene, and heavy oil at single levels.
Carbon Range Standard (C10 to C28)
Ten Sample Analyses
Continuing Calibration Verification (CCV)
RL Standard (Daily) (If required)
Ten Sample Analyses
CCV

10.3.1 The sample extract is reviewed for the presence of the target analytes.

10.3.2 If the concentration of diesel or the site-specific product exceeds the linear range (that is, the highest level standard included in the initial calibration), the sample is diluted and reanalyzed. The dilution should be made to bring the area of the peak of interest into the upper half of the calibration curve. For the single point calibration products, the extract should be diluted until the area is no more than a factor of five above the area of standard.

10.4 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SL SOP AN66: Determination of Retention Time Windows for Gas Chromatographic Analyses.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative analysis

11.1.1 Identification and quantitation of sample concentration is based on the total area (including baseline integration) of all the peaks in the retention time range defined by the reference petroleum standard. The ability to make a positive identification of a specific petroleum product may be inhibited by sample weathering, multi-product composition, and dissimilarity between the sample and the products utilized as standards. Weathered samples will tend to display peaks not found in the reference standard due to compound degradation or may not contain some of the peaks found in the reference standard. Highly weathered samples may also display an elevated baseline resembling a very broad peak or "humpogram." The total area contained in this peak should be used in the quantitative determination if the peak or hump resides within the retention time range of the reference petroleum product.

When product identification is requested, qualitative analysis is limited to the laboratory's standard materials for diesel fuel, mineral spirits, kerosene and motor oil. Other products may be analyzed for at the specific direction of the client. In the absence of such direction, only the above mentioned 4 standard materials are used.

11.1.2 If an identification is not possible, additional information may be provided by the analysis of the straight chain alkane standard. Based on the retention time of the various alkanes, an estimated hydrocarbon chain length for the major constituents in the sample can be identified. The sample chromatogram may be quantified against the petroleum standard that most resembles the sample product and reported with a qualifier such as :

"The peaks in Sample X elute between C10 and C24 and the sample was quantified against the diesel standard, which contains peaks in approximately the same carbon range."

11.1.3 The sample extract must be diluted to point where the peaks are within the linear range of the diesel or site-specific product or to a point where the total area is within a factor of five of the total area of the target products. Diluting the extract into the proper range will aid in the identification and evaluation of the chromatogram.

11.1.4 If DRO is requested, the peaks eluting in the range from C10 to C28 are summed and quantified against the average response factor or curve of the diesel calibration.

11.2 Calculations

11.2.1 Aqueous/Liquid Samples

$$C_{samples}(mg/L) = RF \times area_{sample} \times QF$$

where

RF = response factor

QF = "quant factor"
$Area_{sample}$ = total area of target fuel or compound

This equation expands to:

$$C_{sample}(mg/L) = \frac{mg}{(area)(mL)} \times area_{sample} \times \frac{F \times DF}{V_{ext}}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V_{ext} = volume of sample extracted (L)

If the calibration curve option is used, the sample concentration is calculated:

$$C_{sample} = C_{curve} \times QF$$

where
C_{curve} = concentration of analyte from curve (mg/mL)
QF = "quant factor" (defined above)

11.2.2 Soils/Solids Samples

$$C_{sample}(mg/kg,dw) = RF \times area_{sample} \times QF$$

where
RF = response factor
QF = "quant factor"
$Area_{sample}$ = total area of target fuel or compound

This equation expands to:

$$C_{sample}(mg/kg,dw) = \frac{mg}{(area)(mL)} \times area_{sample} \times \frac{F \times DF}{W \times solids}$$

where
F = final volume of the extract (mL)
DF = dilution factor
W = weight of the sample extracted (kg)
solids = (percent solids)/100

If the calibration curve option is used, the sample concentration is calculated:

$$C_{sample}(mg/kg,dw) = C_{curve} \times QF$$

where
C_{curve} = concentration of analyte from curve (mg/mL)

$$\%REC = \frac{(C_{MS} - C_{sample})}{C_T} \times 100$$

QF = "quant factor" (defined above)

11.2.3 Accuracy is reported as percent recovery, which is calculated:
where
C_{MS} = concentration of matrix spike or lab spike determined from the analysis
C_T = theoretical concentration of the spike

For lab spikes, assume that $C_{sample} = 0$

The theoretical concentration of the spike is calculated:

LIQUIDS

$$C_T = \frac{C_S \times V_S}{V_{sample}}$$

where

C_S = concentration of spiking solution (mg/mL)
V_S = volume of spiking solution added to sample (mL)
V_{sample} = volume of sample extracted

SOLIDS

$$C_T = \frac{C_S \times V_S}{W_{sample} \times solids}$$

where C_S = concentration of spiking solution (mg/mL)
V_S = volume of spiking solution added to sample (mL)
W_{sample} = weight of sample extracted (kg)
solids = (percent solids)/100

11.2.4 Precision is reported as relative percent difference, which is calculated:

$$\%RPD = \frac{|\%REC_{MS} - \%REC_{MSD}|}{(\%REC_{MS} + \%REC_{MSD})/2} \times 100$$

where

$\%REC_{MS}$ = percent recovery of the matrix spike (or lab spike)
$\%REC_{MSD}$ = percent recovery of the matrix spike duplicate (or lab spike duplicate)

12. 0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 SL SOP AN02: *Analytical Batching* describes the procedures for evaluating batch-specific QC. This criteria is summarized in the 8015-Extractable SOP Summary included in this SOP.

12.2 See SL SOP CA90:*Procedure for Determination of Method Detection Limit(MDL).*

13.0 PREVENTIVE MAINTENANCE

No items are included in this revision.

14.0 TROUBLESHOOTING

No items are included in this revision.

15.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

2. Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*, current revisions.

8015-EXTRACTABLES METHOD SUMMARY

The primary targets for this analysis are diesel, mineral spirits, kerosene, and heavy oil. A carbon range standard is analyzed to help the analyst identify the patterns.

HOLD TIMES

MATRIX	Preservative/ storage*	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days	40 days

*Storage temperature is 4C with a control criteria of less than 6C with no frozen samples

EXTRACTION

Aqueous-1-L of sample; continuous or separatory funnel extraction at pH<2 with methylene chloride; concentrate to a nominal final volume of 1.0mL

Soils-30g of sample mixed with sodium sulfate; sonication or Soxhlet extraction with 1:1; methylene chloride/acetone; concentrate to a nominal final volume of 1.0mL

SEQUENCE

STANDARD/SAMPLES
Initial Calibration-diesel at 5 levels; mineral spirits, kerosene, and heavy oil at single levels
Carbon range standard (C10 to C28)
Ten sample analyses
Continuing calibration verification (CCV)
RL standard(optional; required by state or client QAP)
Ten sample analyses
CCV

Sequence continues until all samples have been analyzed or the CCV fails the acceptance criteria

SURROGATE(S):

2-Fluorobiphenyl and o-terphenyl-50ug/L(aqueous); 1700ug/kg(soils)

BATCH QC

Method blank

LCS-diesel at 1mg/ml*

MS/MSD- diesel at 1mg/ml*

*(or site specific target at 5-10X RL)

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration- 5 point minimum with lowest point at RL	Prior to sample analysis or when CCV fails	1) RSD of each target <= 20%; OR 2) plot regression curve	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification(CCV)	After every ten sample analyses and at the end of the sequence	Percent difference or drift <= 15%	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in Table 5 of the SL CQAP	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP
Lab control sample (LCS)- subset of targets in SL CQAP	Per batch (If MS/MSD cannot be performed , the LCS is analyzed)	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP
Matrix spike(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP

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QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Surrogates	All samples, method blanks, and QC	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP
Reporting limit(RL) standard-lowest level calibration standard	Daily(optional-see specific state or client requirements for frequency)	Detected with reasonable sensitivity	-Reanalyze RL standard -Remake and reanalyze RL standard -Perform instrument or column maintenance, recalibrate, and reanalyze associated samples
Retention time window determination	See guidance in SL SOP AN66	See guidance in SL SOP AN66	Use guidance in SL SOP AN66: *Determination of Retention Time Windows in Gas Chromatographic Analyses*
Initial demonstration of Capability	Per analyst	Within the 8000-series method limits (see 8000B)	-Reanalyze QC sample for the targets that failed to meet the criteria
Method detection limit(MDL)	See CA90	Evaluate data according to SL SOP CA90	-Evaluate data according to SL SOP CA90.

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TABLE 1

Petroleum Product	Approximate Carbon Range	Chromatogram Page
Diesel	C10-C24	A, C, D, I, J
Heavy Oil (motor oil or oil range organics)	C20-C34	B, C, D, E, F
Mineral spirits	C8-C12	C, D
Kerosene	C8-C14	C, D
"Weathered" Diesel (50%)	C14-C24	I, J

TABLE 2

Petroleum Product	Approximate Carbon Range	Chromatogram Page
Hydraulic Oil	C22-C36	E, F
Transmission Oil	C20-C30	E, F
Mineral Oil	C24-C36	E, F
Jet A	C10-C16	G, H
JP 5	C10-C16	G, H
JP 8	C10-C16	G, H
Naphtha	<C8-C10	K, L
Turpentine	<C10-<C12	K, L