100645

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

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Submitted To:

U.S. Environmental Protection Agency
Chicago, Illinois

Submitted By:

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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)	RLA (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)	RLA (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)
Endrin	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
Endrin	TO4/TO10	75	33-149	0-32	0.0085	0.10
Endrin aldehyde	TO4/TO10	75	10-130	0-86	0.0079	0.10
Endrin 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/m^3)	RLA (mg/m^3)
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/m^3)	RLA (mg/m^3)
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/m^3)	RLA (mg/m^3)
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/m^3)	RLA (mg/m^3)
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	METHOD (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
Lead (GFAA)	OSHA ID-121	99	80-120	0-20	0.14	0.50
Magnesium (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
Endosulfan 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
Carbon 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
Surrogate - 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
3-Nitroaniline	8270(3550)	97/2	10-117	0-50	11	1700
4-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
Fluorene (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)	RLA (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 A (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)

^ 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 Lab

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 tune 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 | 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

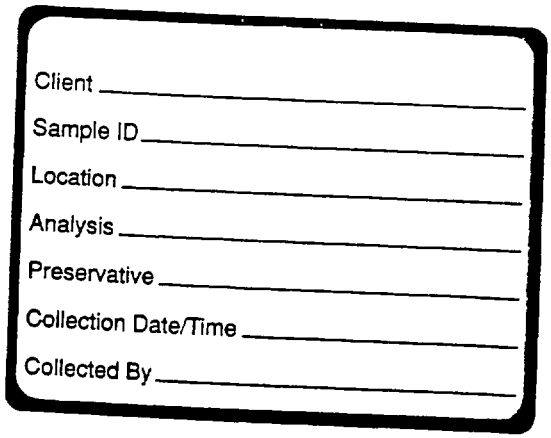

Client
Sample ID
Location
Analysis
Preservative
Collection Date/Time
Collected By

FIGURE 7.3

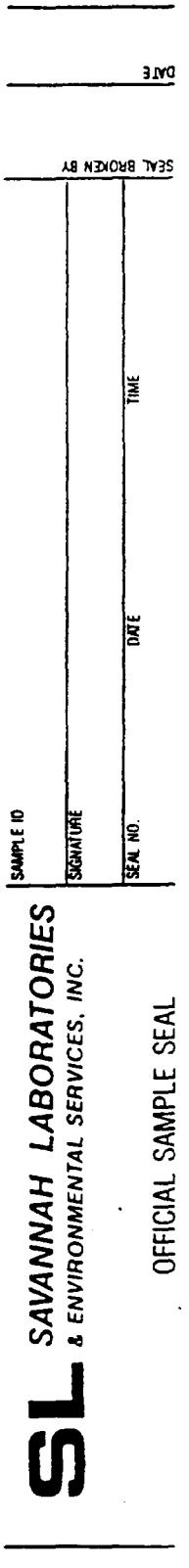

SAMPLE ID
SIGNATURE
SEAL NO.
DATE
TIME
SEAL BROKEN BY
DATE
SL SAVANNAH LABORATORIES
& ENVIRONMENTAL SERVICES, INC.
OFFICIAL SAMPLE SEAL

FIGURE 7.4

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
☐ Custody seals broken
☐ Sample container breakage
☐ Incomplete COC
☐ Cooler temp >6°C or frozen
☐ Sample container partially filled
☐ Sample received not listed on COC
☐ 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

FIGURE 7.5

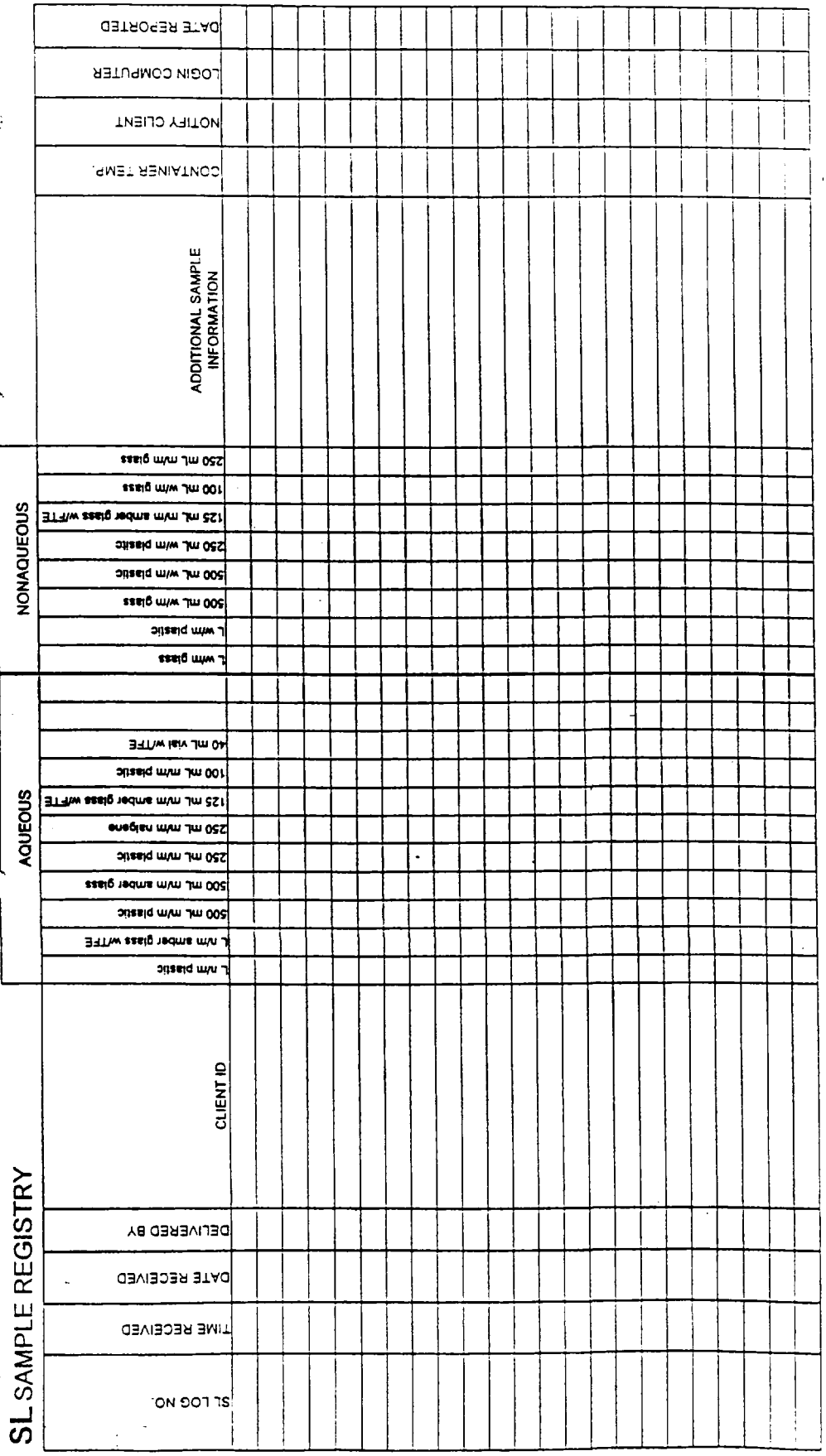

SL SAMPLE REGISTRY
SL LOG NO.
TIME RECEIVED
DATE RECEIVED
DELIVERED BY
CLIENT ID
AQUEOUS
L w/m plastic
L w/m amber glass w/TFE
500 mL w/m plastic
500 mL w/m amber glass
250 mL w/m plastic
250 mL w/m nalgene
125 mL w/m amber glass w/TFE
100 mL w/m plastic
40 mL vial w/TFE
NONAQUEOUS
L w/m glass
L w/m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

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: _____

Phone No: _____

Account No:

Date of Shipment:

Method of Shipment:

Project Reference:

Project Site Location:

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.

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		

FIGURE 7.9

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:

_____

_____

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	

FIGURE 7.10

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

FLUORIDE
Method 340.2

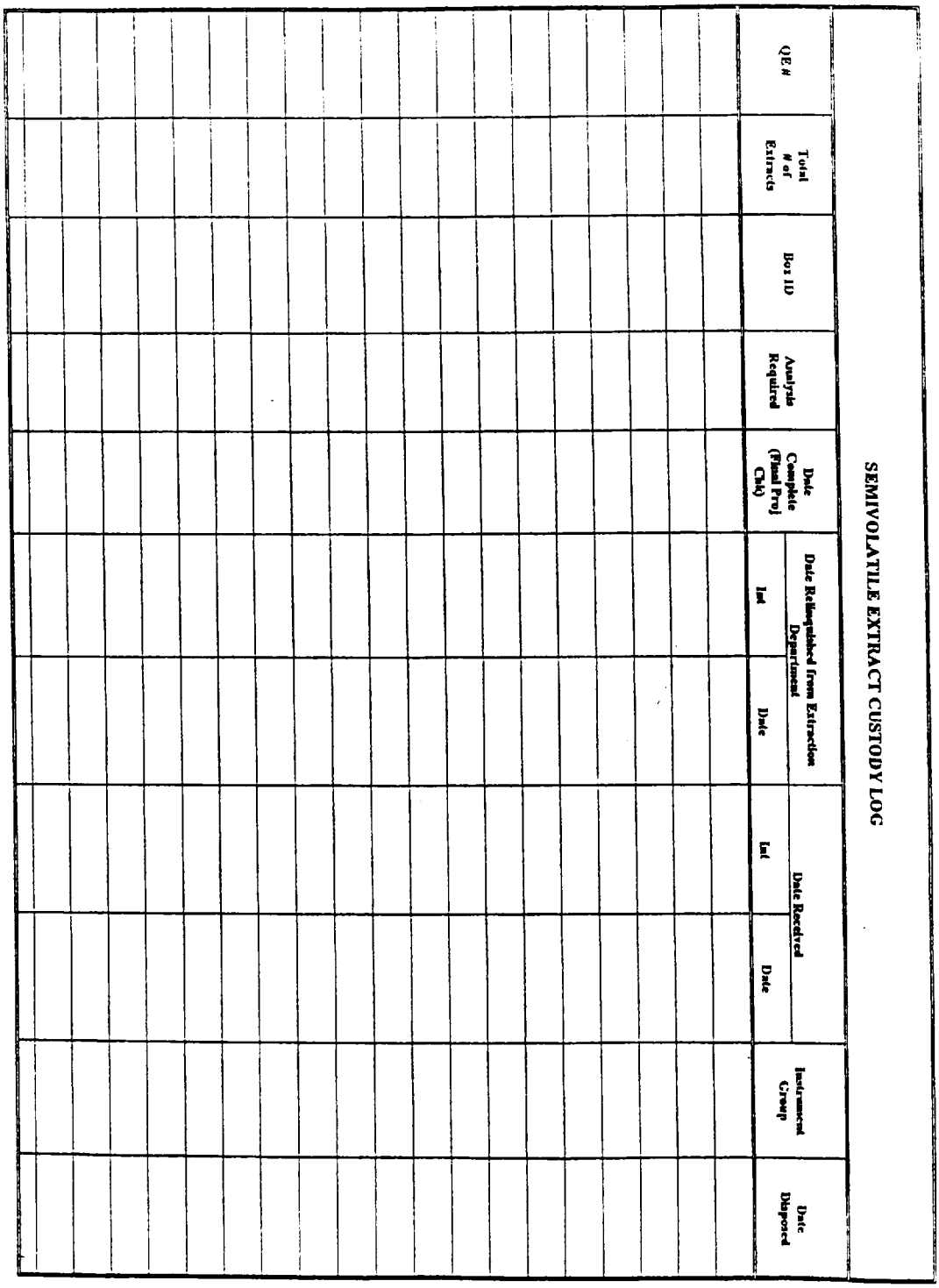
CALIBRATION CURVE		
mg/L	Log (mg/L)	mV
0.1	-1	
0.3	-0.522	
1	0	
2	0.301	
3	0.477	
10	1	
20	1.30	

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 7.12

FIGURE 7.13



FEX005 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 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	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	Room temp Metal case	Used as is.	--	--
		Stock Soln.	Room temp Foil-lined cabinet	Working std, prepped from stock.	Room temp Foil-lined cabinet	As needed
Radon Flask Counter	EPA NIST	Sealed Source	Room temp Foil-lined cabinet	Used as is.	--	--
		Stock Soln.	Room temp Foil-lined cabinet	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	Change.
Purge Flow					X			Inspect semi-annually. Adjust as needed.
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 system schedules and prioritizes all work, provides a mechanism for sample tracking, review of reportables and QC data, generation of reports and invoices, and archiving of all reports and associated QC data. The policies and procedures for the LIMS and other computer 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

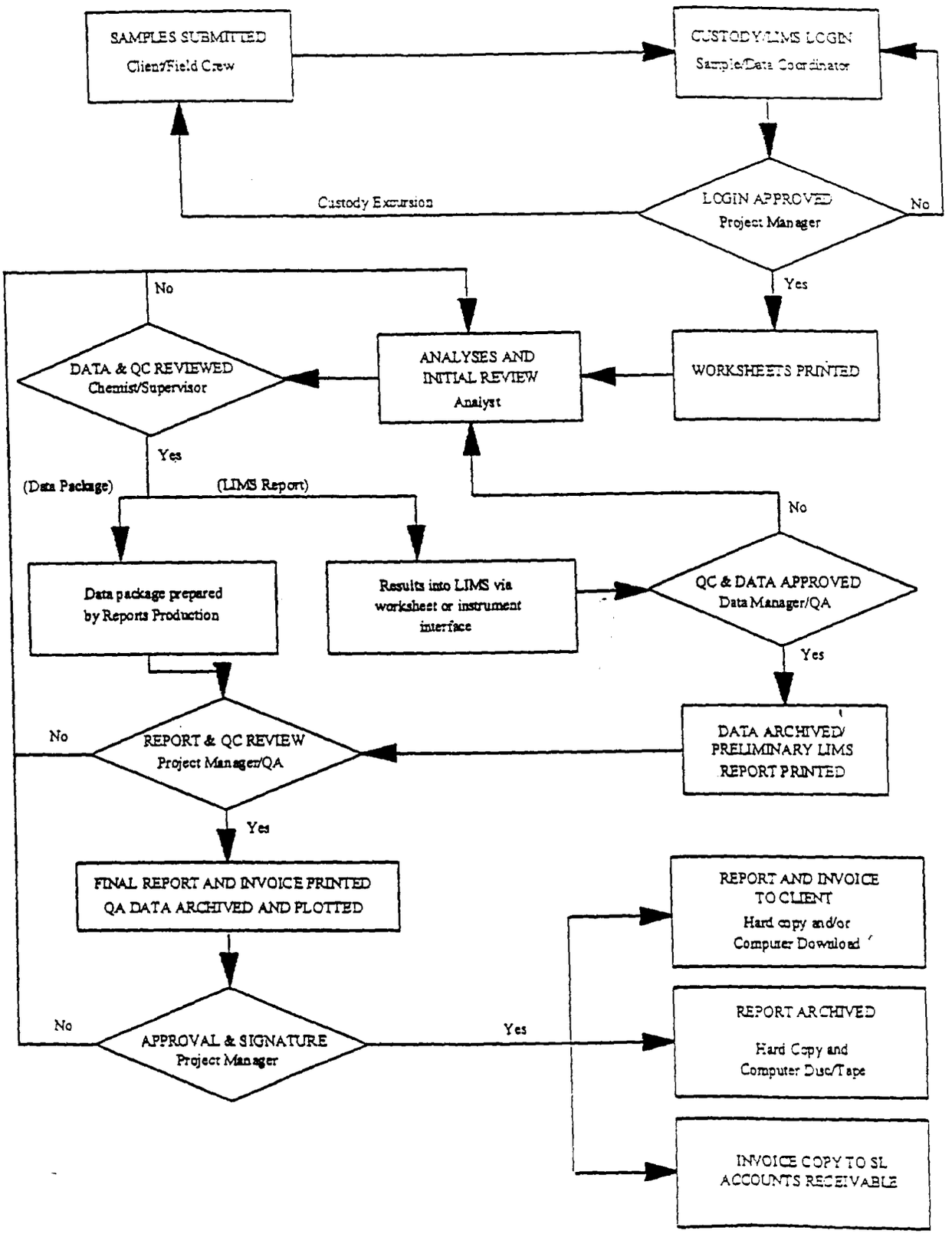

TABLE 12.1

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)

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

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

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

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

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 = $\frac{N_{titrant} \times Titer}{Vol.\ of\ sample\ titrated} \times$ eq. wt. x 1000	mg/L	
BOD	Liquid	
BOD, mg/L = $\frac{(Int.\ DO - Final\ DO) - Seed\ Correction\ Factor}{Vol.\ fraction\ of\ sample}$	mg/L	
C	Liquid	
COD, mg/L = $\frac{(Blk\ titer - sample\ titer) \times N_{FAS} \times 8000}{Vol.\ of\ sample,\ mL}$	mg/L	
Conductivity	Liquid	
Cell constant = $\frac{1000}{Observed\ conductivity\ of\ 1000 - \mu S/cm\ std.}$	µS/cm	
Residue	Liquid	
Residue, mg/L = $\frac{Total\ wt. - Wt.\ of\ dish\ or\ filter}{Vol.\ of\ sample,\ L}$	mg/L	
TOX	Liquid	Solid
TOX, µg/L = $(C_1 + C_2 - 2C_3) \times \frac{1000\ mL}{Vol.\ of\ sample}$ C_1 = instrument reading of 1 column C_2 = instrument reading of 2 column C_3 = instrument reading of blank column TOX, mg/kg = $\frac{instrument\ reading}{\mu L\ injected} \times \frac{5}{dry\ wt.\ fraction}$	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 = $\frac{(\text{cpm sample} - \text{cpm background})\,(1000)}{(2.22)(E_1)(\text{sample volume in mL or sample mass in mg})}$ 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	pCi/L	pCi/g
Radium 226	**Liquid**	
$Ra\text{-}226 = \frac{C \times zt_3}{(2.22)(E)(V)(1 - e^{-zt_1})(e^{-zt_2})(1 - e^{-zt_3})}$ C = net count rate E = calibration constant for system and scintillation 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 = 1.26×10^{-4} h^{-1} z = decay constant for Radon 222	pCi/L	

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 RP (QC level III/IV only)	

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_____

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

Semivolatiles - GC/MS	Target Compound List[1]	Priority Pollutant List[2]	Appendix IX List[3]
Dimethylaminoazobenzene			X
7,12-Dimethylbenz(a)anthracene			X
3,3'-Dimethylbenzidine			X
alpha,alpha-Dimethylphenethylamine			X
2,4-Dimethylphenol	X	X	X
Dimethyl phthalate	X	X	X
1,3-Dinitrobenzene			X
4,6-Dinitro-2-methylphenol	X	X	X
2,4-Dinitrophenol	X	X	X
2,4-Dinitrotoluene	X	X	X
2,6-Dinitrotoluene	X	X	X
Dinoseb			X
1,4-Dioxane			X
1,2-Diphenylhydrazine		X	
Di-n-octyl phthalate	X	X	X
Ethyl methanesulfonate			X
Fluoranthene	X	X	X
Fluorene	X	X	X
Hexachlorobenzene	X	X	X
Hexacholorobutadiene	X	X	X
Hexachlorocyclopentadiene	X	X	X
Hexachloroethane	X	X	X
Hexachlorophene			X
Hexachloropropene			X
Indeno(1,2,3-cd)pyrene	X	X	X
Isophorone	X	X	X
Isosafrole			X
Kepone			X
Methapyrilene			X
3-Methylcholanthrene			X
Methyl methanesulfonate			X
2-Methylnaphthalene	X		X
2-Methylphenol	X		X
3-Methylphenol			X
4-Methylphenol	X		X
Naphthalene	X	X	X
1,4-Naphthoquinone			X
1-Naphthylamine			X
2-Naphthylamine			X
2-Nitroaniline	X		X

Semivolatiles - GC/MS	Target Compound List[1]	Priority Pollutant List[2]	Appendix IX List[3]
3-Nitroaniline	X		X
4-Nitroaniline	X		X
Nitrobenzene	X	X	X
2-Nitrophenol	X	X	X
4-Nitrophenol	X	X	X
5-Nitro-o-toluidine			X
Nitroquinoline-1-oxide			X
N-Nitrosodibutylamine			X
N-Nitrosodiethylamine			X
N-Nitrosodimethylamine		X	X
N-Nitrosomethylethylamine			X
N-Nitrosodiphenylamine/diphenylamine	X	X	X
N-Nitrosodi-n-propylamine	X	X	X
N-Nitrosomorpholine			X
N-Nitrosopiperidine			X
N-Nitrosopyrrolidine			X
Pentachlorobenzene			X
Pentachloronitrobenzene			X
Pentachlorophenol	X	X	X
Phenacetin			X
Phenanthrene	X	X	X
Phenol	X	X	X
1,4-Phenylenediamine			X
2-Picoline			X
Pronamide			X
Pyrene	X	X	X
Pyridine			X
Safrole			X
1,2,4,5-Tetrachlorobenzene			X
2,3,4,6-Tetrachlorophenol			X
o-Toluidine			X
1,2,4-Trichlorobenzene	X	X	X
2,4,5-Trichlorophenol	X		X
2,4,6-Trichlorophenol	X	X	X
1,3,5-Trinitrobenzene			X
O,O,O-Triethyl phosphorothioate			X

Chlorinated Dioxins and Furans Method 8280	Target Compound List[1]	Priority Pollutant List[2]	Appendix IX List[3]
Tetrachlorodibenzo-p-dioxins			X
Tetrachlorodibenzofurans			X
Pentachlorodibenzo-p-dioxins			X
Pentachlorodibenzofurans			X
Hexachlorodibenzo-p-dioxins			X
Hexachlorodibenzofurans			X
2,3,7,8-Tetrachlorodibenzo-p-dioxin		X	X

Chlorinated Pesticides-GC	Target Compound List[1]	Priority Pollutant List[2]	Appendix IX List[3]
Aldrin	X	X	X
alpha-BHC	X	X	X
beta-BHC	X	X	X
gamma-BHC	X	X	X
delta-BHC	X	X	X
Chlordane		X	X
alpha-Chlordane	X		
gamma-Chlordane	X		
Chlorobenzilate			X
4,4'-DDD	X	X	X
4,4'-DDE	X	X	X
4,4'-DDT	X	X	X
Dieldrin	X	X	X
Endosulfan I	X	X	X
Endosulfan II	X	X	X
Endosulfan sulfate	X	X	X
Endrin	X	X	X
Endrin aldehyde	X	X	X
Endrin ketone	X		
Heptachlor	X	X	X
Heptachlor epoxide	X	X	X
Isodrin			X
Methoxychlor	X		X
Toxaphene	X	X	X

PCBs-GC	Target Compound List[1]	Priority Pollutant List[2]	Appendix IX List[3]
Aroclor-1016	X	X	X
Aroclor-1221	X	X	X
Aroclor-1232	X	X	X
Aroclor-1242	X	X	X
Aroclor-1248	X	X	X
Aroclor-1254	X	X	X
Aroclor-1260	X	X	X

Chlorinated Herbicides Method 8151	**Target Compound List[1]**	**Priority Pollutant List[2]**	**Appendix IX List[3]**
2,4-D			X
2,4,5-T			X
2,4,5-TP Silvex			X

Organophosphorus Pesticides Method 8141	**Target Compound List[1]**	**Priority Pollutant List[2]**	**Appendix IX List[3]**
Disulfoton			X
Methyl Parathion			X
Ethyl Parathion			X
Tetraethyl dithiopyrophosphate (sulfotepp)			X
Famphur			X
Phorate			X
o,o-Diethyl-o-2-pyrazinyl phosphotothio			X
Dimethoate			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.

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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STANDARD OPERATING PROCEDURES
Table of Contents

Category	SOP Number	SOP Name	Document Control Number
Air	AR05	Analysis of Whole Air Samples in Tedlar Bags	AR05:05.01.98:6
	AR10	Whole Air Analysis of VOC in Summa-Passivated Canisters	AR10:02.06.98:1
	AR15	SVOC in Ambient Air-BNA and Pesticides Collected on PUF and PUF/Resin Cartridges(TO4/TO10/TO13)	AR15:01.21.98:1
	AR30	Dissolved Gases in Water	AR30:08.11.97:3
Ancillary	AN01	Standard Operating Procedure for the Generation of SOP's	AN01:08.07.98:3
	AN02	Analytical Batching	AN02:05.18.98:2
	AN10	Balance Calibration and Use	AN10:08.29.97:3
	AN15	Tekmar Sonic Dismembrator Tuning Procedures	AN15:08.26.97:1
	AN20	Refrigerated Storage Space Temperature Checks	AN20:08.26.97:2
	AN22	Desiccator Checks Maintenance and Use	AN22:12.03.97:0
	AN30	Pipet and Volumetric Container Calibration Check	AN30:04.23.98:3
	AN35	Conductivity Checks for Laboratory Deionized Water	AN35:08.26.97:3
	AN41	Standard Materials Traceability	AN41:10.14.97:2
	AN43	Standard Preparation	AN43:04.23.98:0
	AN44	Reagent Traceability	AN44:05.18.98:1
	AN45	Laboratory Notebooks	AN45:03.04.98:4
	AN50	Wavelength Check for Spectrophotometers	AN50:08.26.97:1
	AN51	Installation and Evaluation of New Instrument Systems	AN51:05.22.97:0
	AN52	Test Procedures for Method Development and Modification	AN52:05.22.97:0
	AN55	Laboratory Thermometer Calibration	AN55:08.26.97:2
	AN60	Glassware Cleaning Procedures	AN60:08.26.97:1
	AN60-M	Glassware Cleaning Procedure-MOBILE DIVISION	AN60-M-1:11.10.97:1

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Category	SOP Number	SOP Name	Document Control Number
Ancillary	AN65	Manual Integrations	AN65:04.23.98:1
	AN66	Determination of Retention Time Windows for Gas Chromatographic	AN66:02.25.97:1
	AN70	Sample Compositing, Homogenization, and Segregation of Low/High Concentration VOA/SVOA	AN70:08.11.98:2
	AN75	LIMS Control Chart System	AN75:07.14.98:1
	AN80	Sterilization of Foreign Soil Samples	AN80:04.23.98:3
	AN90	Project Initiation	AN90:06.24.97:1
	AN91	Procedure for the Generation of Electronic Deliverables via the LIMS	AN91:02.19.97:0
	AN92	Procedure for Data/system Backup on HP9000 Chemservers	AN92-S:04.24.97:0
	AN93	Procedure for Disaster Recovery on HP9000 Chemservers	AN93-S:04.24.97:0
	AN96	LIMS System Modification and Testing	AN96-S:04.30.98:1
	AN97	LIMS System Maintenance	AN97-S:05.19.97:0
	AN98	LIMS System Security	AN98-S:05.19.97:0
Corporate Administrative	CA01	SL Training SOP	CA01:05.05.97:5
	CA02	Divisional Document Control and Distribution	CA02:04.08.97:1
	CA03	Contract Review	CA03:05.30.98:0
	CA05	Technical and Systems Audits (Divisional and Corporate)	CA05:05.14.97:0
	CA10	Procedures for New Employee Orientation	CA10:04.14.97:0
	CA20	Telephone Answering Procedures	CA20:05.01.98:1
	CA25	Stop Work Authority	CA25:05.01.98:1
	CA45	Corporate Procurement	CA45:02.27.98:1
	CA50	Laboratory Certifications	CA50:07.15.97:0
	CA70	Waste Disposal	CA70:01.30.97:0
	CA80	Data Generation, Entry, Review, Approval, and Reporting	CA80:09.30.98:6
	CA81	Regeneration of Deliverable Reports	CA81:09.30.98:0

Category	SOP Number	SOP Name	Document Control Number
Corporate Administrative	CA85	Nonconformance and Corrective Action Procedures	CA85:08.07.98:3
	CA90	Procedure for the Determination of Method Detection Limits (MDL)	CA90:10.29.97:0
	CA91	Determination of the Instrument Detection Limit (IDL)	CA91:09.04.97:0
	CA92	Evaluation of IDOC's	CA92:08.21.98:0
	CA95	Complaint Resolution	CA95:06.16.97:1
	CA96	Project Management	CA96:11.19.97:0
	CA97	Evaluation of Laboratory Capacity and Capabilities for Accepting New Projects and Analyses/Analytes	CA97:12.23.97:0
	CA98	Analytical Sub-Contractors Policy	CA98:04.27.98:0
Custody	CU01	Receipt, Log Number Assignment, and Distribution of Field Samples	CU01:04.27.98:1
	CU02-S	Internal Chain-Of-Custody	CU02-S:07.10.98:0
	CU15	Preparation of Sample Containers	CU15:06.19.98:3
	CU16	Wipe Test: Sampling	CU16:07.25.97:0
	CU20	Interlaboratory Sample Exchange	CU20:03.31.98:1
	CU35	Procedures for Contaminant-Free Sample Containers	CU35:06.19.98:1
Extractions	EX10	EP Toxicity Extraction	EX10:05.01.98:1
	EX15	TCLP Extraction: Nonvolatiles	EX15:08.04.97:2
	EX16	TCLP Extraction: Volatiles	EX16:08.05.97:1
	EX17	SPLP Extractions: Semivolatile Organic Compounds, Metals, and Cyanide	EX17:03.23.98:0
	EX18	SPLP Extractions: Volatiles	EX18:03.23.98:0
	EX25	Extraction Procedure for Oily Waste	EX25:05.01.98:1
	EX30	Continuous Liquid-Liquid Extraction	EX30:02.19.98:1
	EX35	Separtory Funnel Extraction	EX35:08.04.97:0
	EX40	Ultrasonic Extraction	EX40:02.19.98:1
	EX42	Waste Dilution Extraction	EX42:07.06.98:0

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Category	SOP Number	SOP Name	Document Control Number
Extractions	EX45	Extraction OF Chlorinated Herbicides in Water, Soils, and Wastes	EX45:08.29.97:1
	EX50	Zymark Extraction Concentration Procedure	EX50:08.04.97:0
	EX51	Kuderna-Danish Extract Concentration Procedure	EX51:08.04.97:0
	EX60	Acid, Permanganate, and Copper Cleanups for PCBs and Pesticides	EX60:10.22.97:0
	EX63	Silica Gel Cleanup and Fractionation	EX63:02.17.99:1
	EX70	Preparation of SVOA Surrogate and Matrix Spiking Solutions	EX70:09.02.97:1
FD	FD05	Monitoring Well Sampling Procedure	FD05:05.01.98:1
General	BA05	Total Organic Carbon in Soil: Walkley-Black Method	BA05:08.26.97:2
	BA09	Total Organic Carbon-Shimadzu TOC Analyzer	BA09:05.01.98:1
	BA11-T	Total Organic Carbon: Dohrmann DC-80 Method	BA11:05.01.98:2
	BA12	Total Organic Halides (TOX)	BA12:03.26.98:2
	BA13	Extractable Organic Halides (EOX)	BA13:02.23.98:1
	BA14	Absorbable Organic Halides (AOX)	BA14:05.13.98:3
	BA15	Biochemical Oxygen Demand	BA15:10.07.98:3
	BA20	Chemical Oxygen Demand (Titrimetric Procedure)	BA20:08.05.97:2
	BA22	Chemical Oxygen Demand (Hach Method)	BA22:01.01.96:0
	BA25	Coliforms: Membrane Filter Technique	BA25:05.01.98:1
	BA30	Coliforms: Multiple Tube Technique	BA30:05.01.98:1
	BA31	Coliforms in Potable Water: Colilert P/A Method	BA31:05.01.98:1
	BA35	Alkalinity (and Other Carbonate Species)	BA35:12.16.98:3
	BA36	Acidity	BA36:12.15.98:3
	BA37	Corrosivity (Langlier Saturation Index)	BA37:05.01.98:2
	BA40	Total Dissolved Solids and Volatile Dissolved Solids	BA40:08.26.97:2
	BA45	Total Suspended Solids and Volatile Suspended Solids	BA45:08.26.97:1

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Category	SOP Number	SOP Name	Document Control Number
General	BA46	Total Residue and Volatile Solids	BA46:08.26.97:0
	BA50	Total solids, Ash, and Volatile Solids in Soils, Solids, and Semisolids	BA50:08.26.97:4
	BA51	Percent Solids/Percent Moisture for Reporting Soils on a "Dry-Weight" Basis	BA51:08.26.97:0
	BA55	Settleable Matter	BA55:02.16.98:2
	BA60	Paint Filter Liquids Test	BA60:05.01.98:2
	BA65	Turbidity	BA65:05.01.98:2
	BA70	pH Electrometric Measurement of Water, Soil, and Waste	BA70:03.06.97:1
	BA80	Specific Conductance (Conductivity)	BA80:02.16.98:2
	BA81	Density	BA81:02.16.98:1
	BA85	Dissolved Oxygen	BA85:08.11.98:3
	BA86	Specific Gravity	BA86:05.01.98:1
	BA90	Color	BA90:02.16.98:2
	BA95	Odor	BA95:02.16.98:2
	GE05	Cation Exchange Capacity	GE05:05.01.98:2
	GE10	Heat of Combustion BTU/LB	GE10:03.19.97:1
	GE100	Sulfide	GE100:02.19.98:1
	GE101	Spectrophotometric Determination of Sulfides	GE101:05.12.98:3
	GE102-M	Sulfide, Methylene Blue	GE102:05.01.98:1
	GE105	Total Sulfur	GE105:03.19.97:1
	GE115	Ion Chromatography	GE115:05.01.98:3
	GE116-M	Bromide - Titrimetric	GE116:05.01.98:3
	GE120	Chloride: Autoanalyzer Procedure	GE120:02.11.98:2
	GE121-M	Chloride: Lachat Procedure	GE121:05.01.98:2
	GE122	Titrimetric Chloride	GE122:05.12.98:0

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Category	SOP Number	SOP Name	Document Control Number
General	GE125	Salinity and Percent Seawater	GE125:05.01.98:1
	GE130	Fluoride	GE130:05.01.98:3
	GE131	Total Residual Chlorine DPD Method 8167	GE131:05.01.98:1
	GE132	Sulfite: Method 4500-B	GE132:05.01.98:1
	GE133	Total Residual Ch;prome by Iodometric Titration	GE133:12.15.98:0
	GE140	Ignitability	GE140:05.01.98:1
	GE15	Ammonia: Lachat QuikChem 8000 Autoanalyzer Procedure	GE15:08.13.98:1
	GE150	Oil and Grease and Petroleum Hydrocarbons by IR	GE150:02.09.98:3
	GE155	Oil and Grease by Gravimetry	GE155:05.01.98:2
	GE157	Oil and Grease and Petroleum Hydrocarbons by Gravimetry	GE157:01.14.98:0
	GE16-M	Ammonia - Lachat Autoanalyzer Method	GE16:05.01.98:1
	GE160	Methylene Blue Active Substances	GE160:12.15.98:2
	GE165	Total Recoverable Phenolics: Manual Procedure	GE165:05.01.98:2
	GE175	Water in Nonaqueous Samples by Distillation	GE175:05.01.98:1
	GE18	Calculation of Un-ionized Ammonia in Fresh Water	GE18:03.25.98:0
	GE185	Determination of Nitrocellulose in Water	GE185:05.01.98:1
	GE186	Determination of Nitrocellulose in Soil	GE186:05.01.98:1
	GE20	Block Digestion of Samples for Semiautomated Determination of Total Kjeldahl Nitrogen and Total	GE20:10.14.98:4
	GE26-M	Kjeldahl Nitrogen and Total Phosphorus: Lachat QuickChem AE Procedure	GE26:05.01.98:1
	GE27	Calculation of Total and Organic Nitrogen	GE27:12.15.98:0
	GE31	Nitrate Plus Nitrite: Lachat QuickChem AE Procedure	GE31:05.01.98:1
	GE36	Nitrite-N: Lachat QuickChem AE Procedure	GE36:05.01.98:1
	GE40	Total and Amenable Cyanide: Autoanalyzer Procedure	GE40:02.02.98:3
	GE41	Total Cyanide: Autodistillation Procedure	GE41:05.01.98:3

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Category	SOP Number	SOP Name	Document Control Number
General	GE43	Midi Distillation of Water and Soils for the Determination of Cyanide	GE43:02.18.98:1
	GE46-M	Total Cyanide and Cyanide Amenable to Chlorination by Manual Distillation	GE46-M:03.05.96:0
	GE55	Hydrogen Cyanide Released from Wastes (Reactive Cyanide and Sulfide)	GE55:08.05.97:4
	GE60	Acid-Hydrolyzable Phosphorus	GE60:05.01.98:1
	GE65	Available Phosphorus in Soils	GE65:05.01.98:1
	GE71	Orthophosphate: Manual Colorimetric	GE71:08.07.98:0
	GE75	Phosphorus Adsorption	GE75:05.01.98:4
	GE81	Phosphorous: Lachat Procedure (Acid Persulfate Digestion Procedure)	GE81:05.23.97:1
	GE85	Acid-Soluble Sulfide	GE85:02.19.98:3
	GE90	Acid Volatile Sulfides	GE90:05.12.98:4
	GE96-M	Sulfate (Gravimetric)	GE96:05.01.98:1
	GE97	Turbidimetric Sulfate (375.4/9038)	GE97:05.12.98:0
Liquid Chromatography	LC10-T	Formaldehyde by HPLC	LC10:05.01.98:2
	LC15-T	Carbamate Pesticides by HPLC	LC15:05.01.98:1
	LC16-T	Sample Preparation for Aldicarb and Its Metabolites in Nectar and Soil by HPLC	LC16:05.01.98:1
	LC20-T	Pesticides and Herbicides by HPLC	LC20:05.01.98:1
	LC25-T	N-methyl Carbamate Pesticides in Soils	LC25:05.01.98:1
	LC30	Determination of PAHs in Water and Soil by HPLC	LC30:05.01.98:1
	LC35-T	Thiodiglycol by HPLC	LC35:05.01.98:1
	LC40-T	Determination of Explosive Residues in Water and Soil bby HPLC	LC40-T:10.28.98:4
	LC45-T	Determination of Asulam in Water by HPLC	LC45:05.01.98:1
	LC60-T	Ethylenethiourea In Water and Soils	LC60-T:08.10.98:0
	LC65-T	Phthalic Acid &Maleic Acid in Water & Soil & Phthalic Acid as Phthalic Anhydride in Soil by HPLC	LC65-T:12.18.98:0
Metals	ME20	Hexavalet Chromium Colorimetric Procedure	ME20:08.26.97:1

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Category	SOP Number	SOP Name	Document Control Number
Metals	ME21	Alkaline Digestion of Sediments, Sludges, and Soils for Hexavalent Chromium	ME21:04.06.98:0
	ME22	Hexavalent Chromium-Lachat Autoanalyzer Procedure	ME22:08.06.98:0
	ME26	Mercury: Varian Spectra AA 20	ME26:05.01.98:3
	ME27-M	Mercury -MOBILE	ME27-M:11.17.97:1
	ME28	Mercury Analysis: Leeman PS200	ME28:12.19.97:0
	ME29	Mercury Preparation: Leeman AP200	ME29:12.19.97:0
	ME30-M	Hydride Digestion	ME30:05.01.98:1
	ME36-M	Hydride Generation AA Analysis for As and Se - MOBILE	ME36:05.01.98:1
	ME40-T	MIBK Extraction	ME40-T:10.28.98:2
	ME50	Digestion Procedures for ICP - Total Metals and Total Recoverable Metals in Liquid Samples	ME50:04.01.98:5
	ME51	Digestion Procedures for ICP - Total Metals in Soils and Sediments	ME51:07.02.98:1
	ME52	Parr Bomb Digestion Procedures for ICP	ME52:06.12.97:0
	ME60	Digestion Procedures for GFAA - Total Metals and Total Recoverable Metals in Liquid Samples	ME60:04.01.98:5
	ME61	Digestion Procedures for GFAA - Total Metals in Soils and Sediments	ME61:07.02.98:1
	ME62	Parr Bomb Digestion Procedures for GFAA	ME62:06.27.97:0
	ME70	ICP Operating Procedure	ME70:06.19.98:6
	ME75	Graphite Furnace AA	ME75:12.22.97:3
	ME90	Calculation of Hardness by SM2340B	ME90:08.11.98:0
Project Specific	PS15	Analytical Scheme and Report Deliverable Format for PCBs in Fish and Other Biological Tissues	PS15:02.11.98:2
Quality Control	QC16	Minimum Analytical Records Maintained by SL	QC16:08.26.98:1
Radioactivity	RA01-B	Gross Alpha and Beta Radioactivity	RA01:05.01.98:1
	RA02-B	Radium - 226	RA02:05.01.98:1
	RA03-B	Radium - 228	RA03:05.01.98:1
Report Format	RF05	SL Data Qualifiers	RF05:05.01.98:2

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Category	SOP Number	SOP Name	Document Control Number
Semivolatile Compounds by GC	SG05-T	Dithiocarbamate Pesticides (EPA630)	SG05-T:08.10.98:0
	SG40	Phenols and PAHs by GC	SG40:12.19.97:1
	SG45	Organochlorine Pesticides and PCBs	SG45:07.15.98:5
	SG50	Organophosphorus and Organonitrogen Pesticides	SG50:05.01.98:3
	SG60	1,2-Dibromoethane	SG60:05.01.98:3
	SG65	Chlorinated Herbicides	SG65:01.12.99:4
	SG70	Modified 8015 Extractable for Petroleum Identification	SG70:01.16.98:4
	SG71	Extractable Petroleum Hydrocarbons (EPH)-Massachusetts Method	SG71:05.28.98:0
	SG85-M	Triazine Pesticides and Other Organonitrogen Compounds	SG85:05.01.98:2
	SG90	Nitrosamines by Gas Chromatorgraphy	SG90:05.01.98:1
	SG95	Thiocarbamate Pesticides	SG95:06.26.96:0
Semivolatile Compounds by GC/MS	SM01	Semivolatile Compounds by GC/MS EPA 625	SM01:05.18.98:3
	SM05	Semivolatile Compounds by GC/MS	SM05:12.19.97:5
	SM06	Guidelines for SIM Analyses by GC/MS	SM06:01.20.98:0
	SM10	Polychlorinated Dibenzo-p-dioxin and Polychlorinated Dibenzofurans	SM10:08.18.98:3
	SM30-M	Endothall (Method 548.1) MOBILE	SM30:05.01.98:1
	SM31	Chlorinated Phenolics in Wastewater by In-Situ Acetylation and GC/MS EPA Method 1653	SM31:02.17.98:1
Volatile Compounds by GC	VG05	Volatile Compounds by GC(601/602, 8010/8020, 8015B, 8021)	VG05:07.06.98:4
	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

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Category	SOP Number	SOP Name	Document Control Number
Volatile Compounds by GC	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

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 + ..RFn}{n}$$

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:

$$\%Drift = \frac{result - expected}{expected} \times 100$$

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

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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
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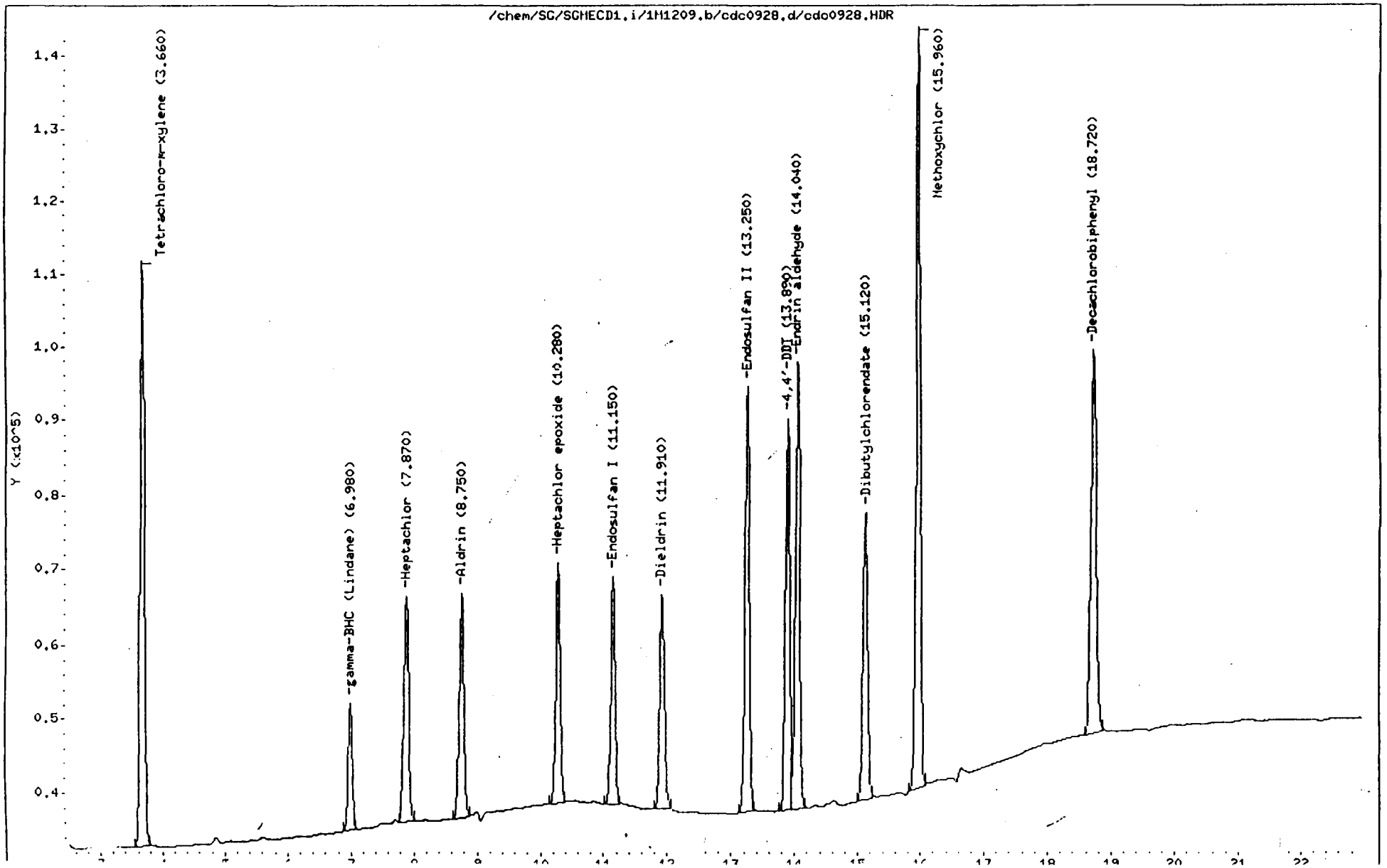
Sample Info: PISHA-04~1H1209

Instrument: SGHECD1.i

Operator: BW
Column diameter: 0.53

Column phase: DB-608



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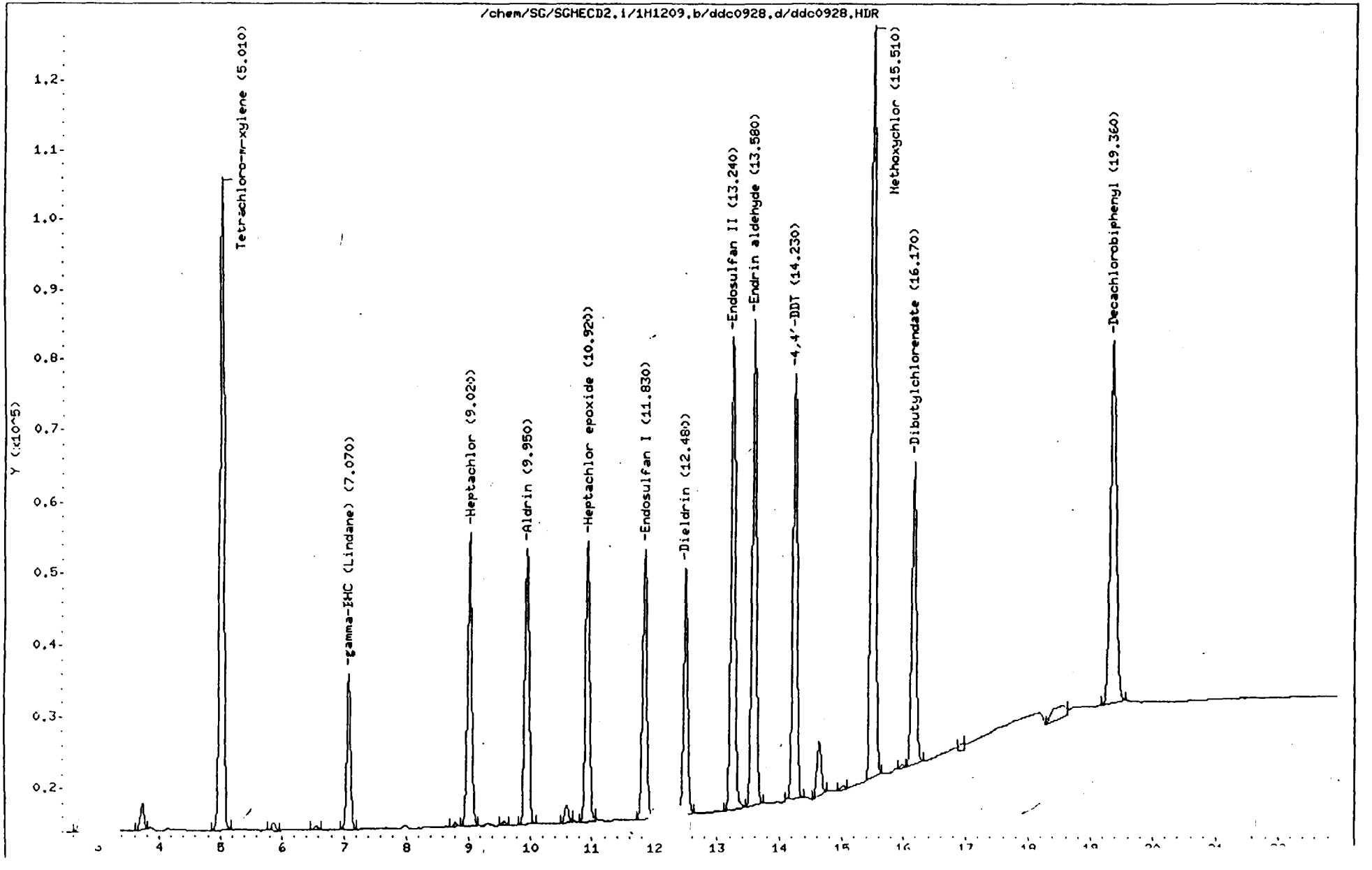
Sample Info: PISHA-04^1H1209

Instrument: SGHECD2.i

Operator: BW
Column diameter: 0.53

Column phase: DB-5



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Sample Info: PISMB-04~1M1209

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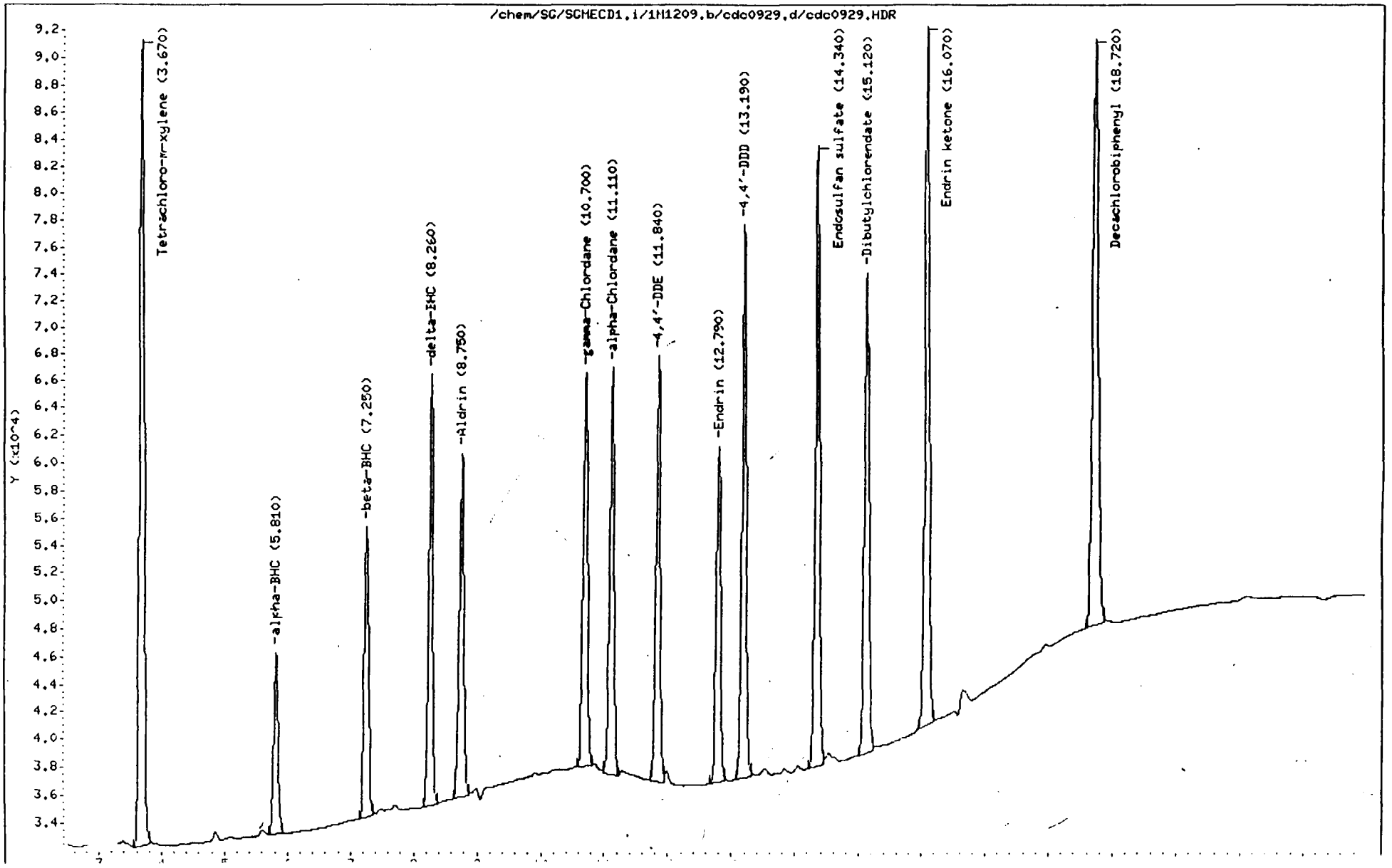
Column diameter: 0.53



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Sample Info: PISHB-04~1H1209

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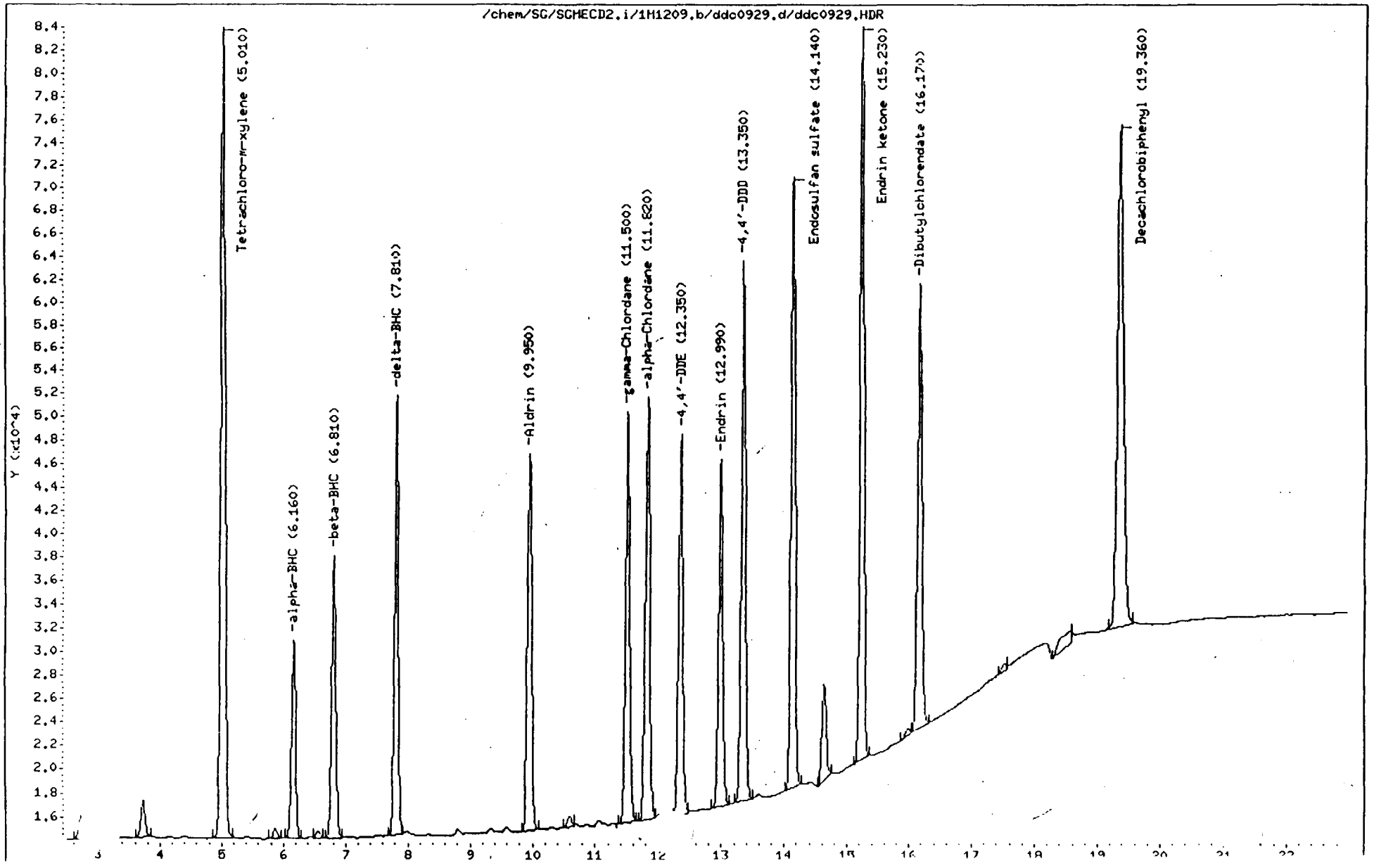
Column phase: DB-5



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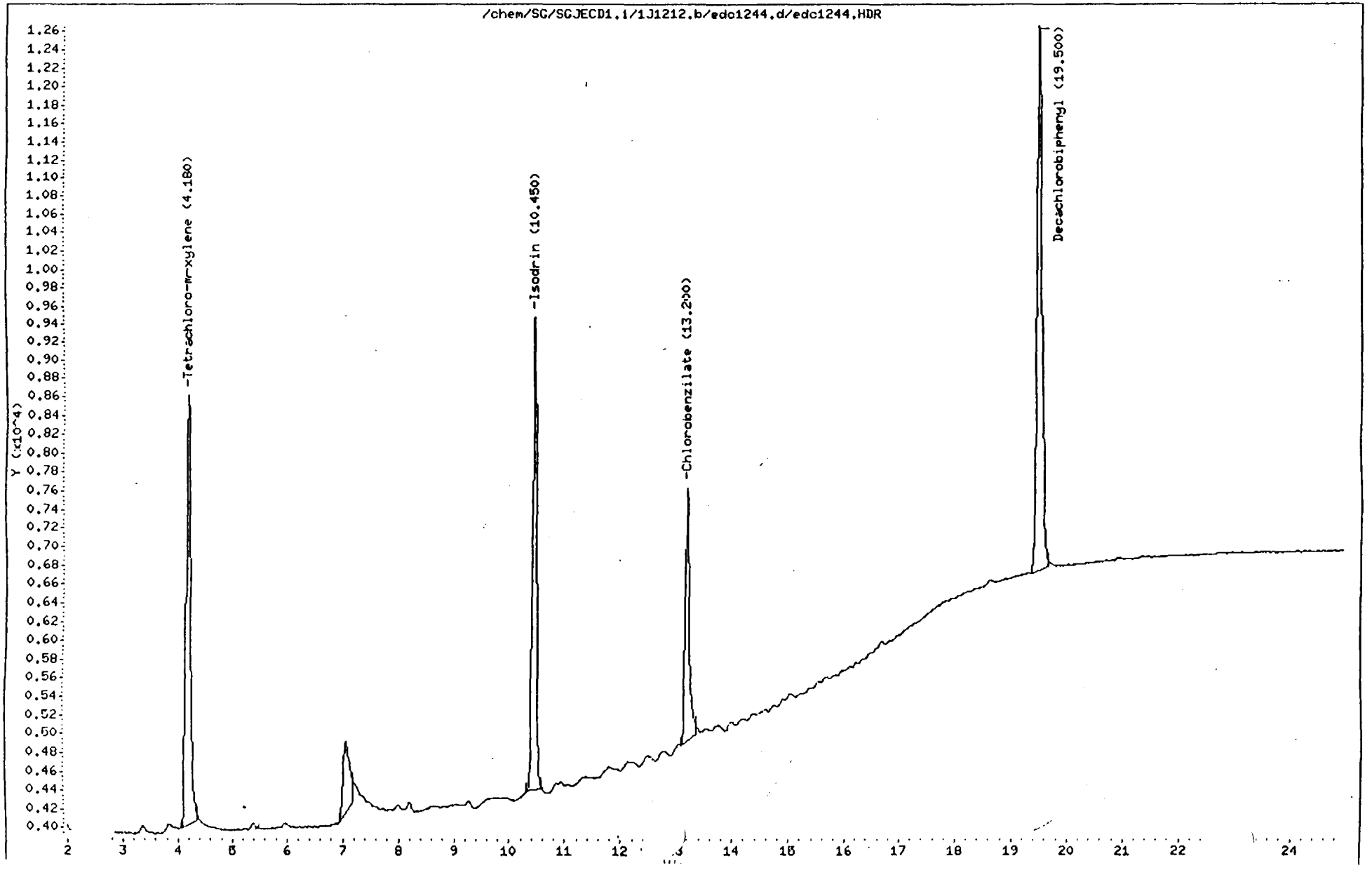
Instrument: SGJECD1.i

Sample Info: PISOC-04~1J1212

Operator: BW

Column phase: DB-608

Column diameter: 0.53



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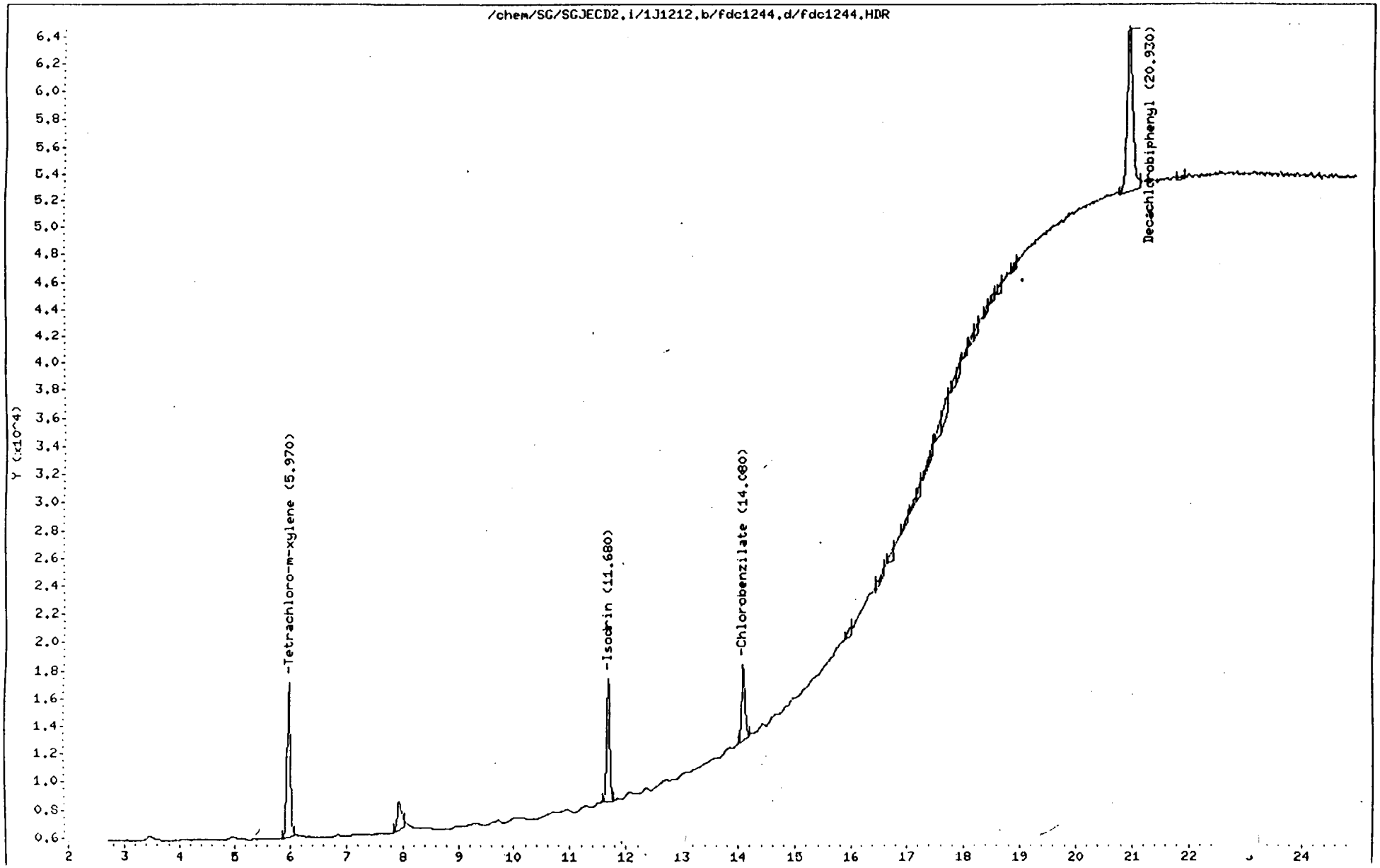

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Sample Info: CHLOR-04~1H1209

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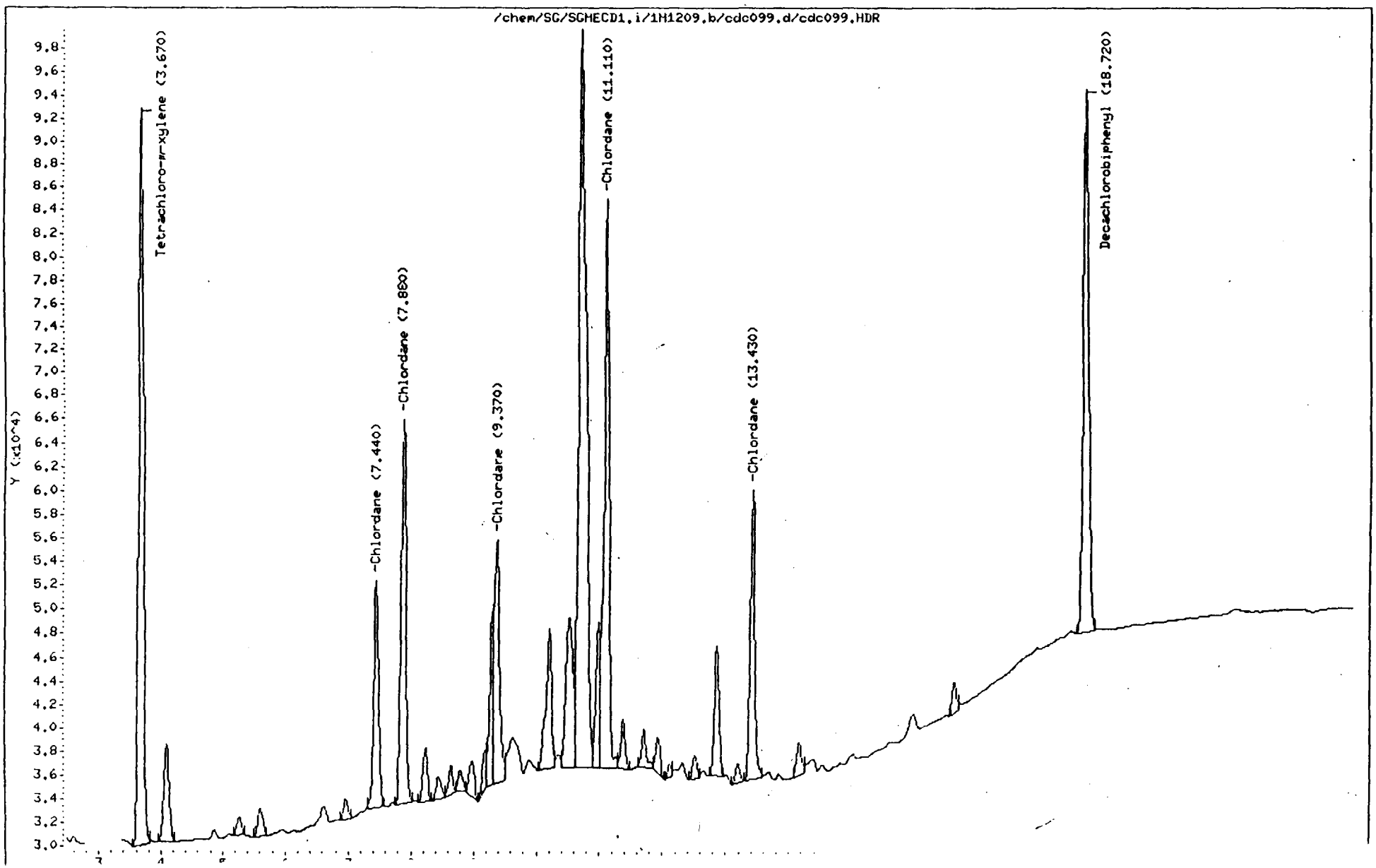
Operator: BW
Column diameter: 0.53

Column phase: DB-608



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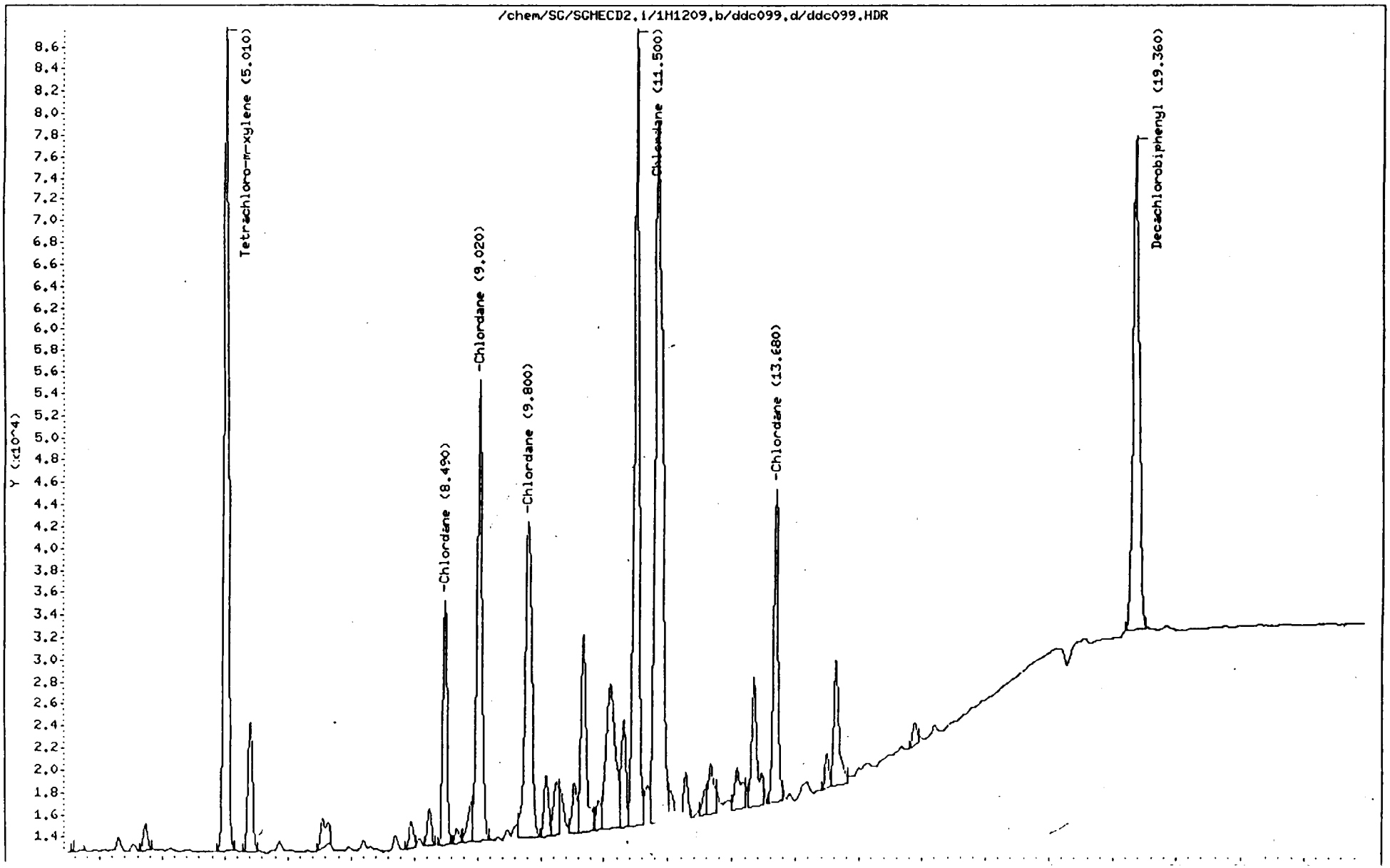
Column phase: DB-5



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Sample Info: TOXAPH-04^1H1209

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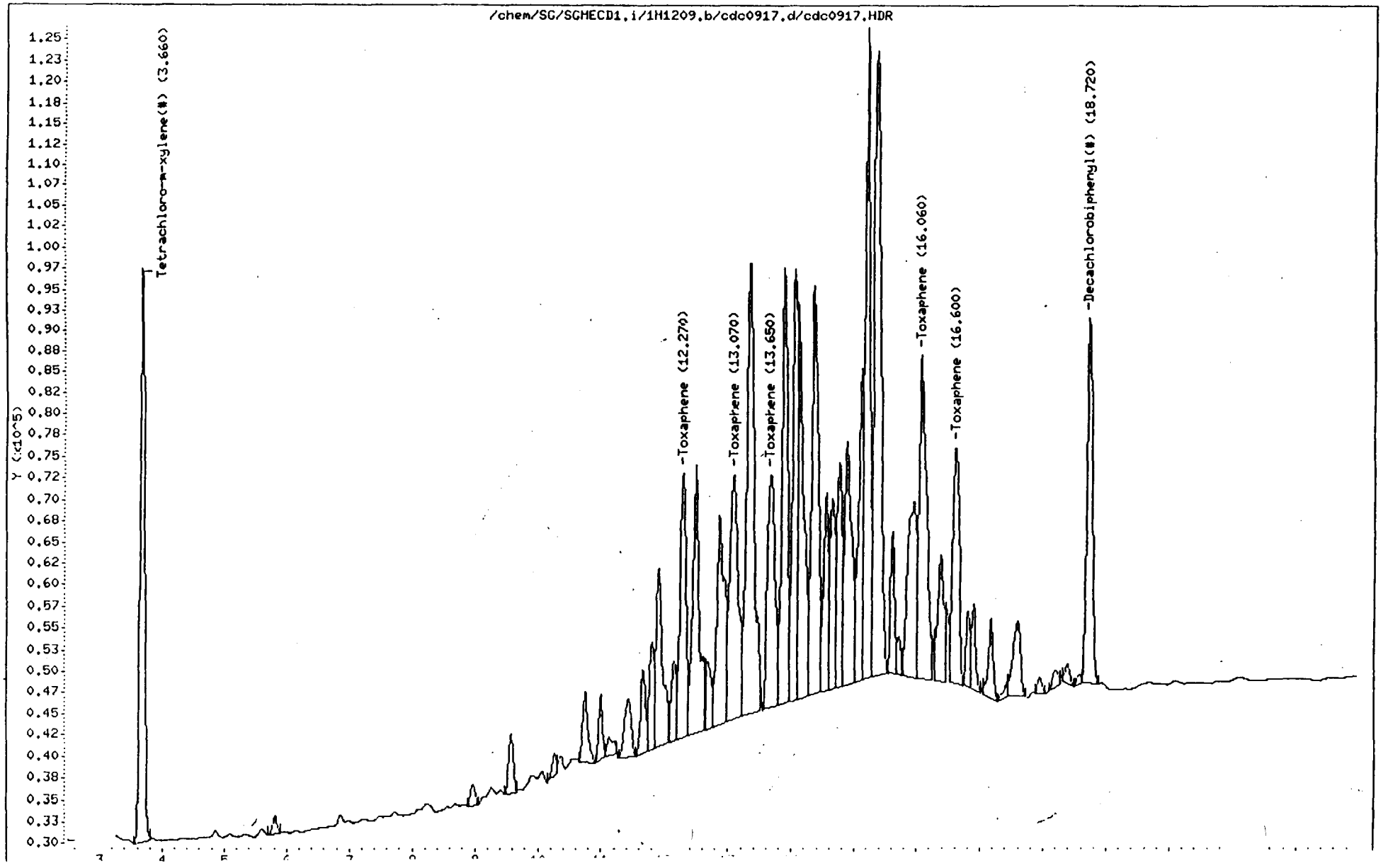
Operator: BW
Column diameter: 0.53

Column phase: DB-608



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Sample Info: TOXAPH-04~1H1209

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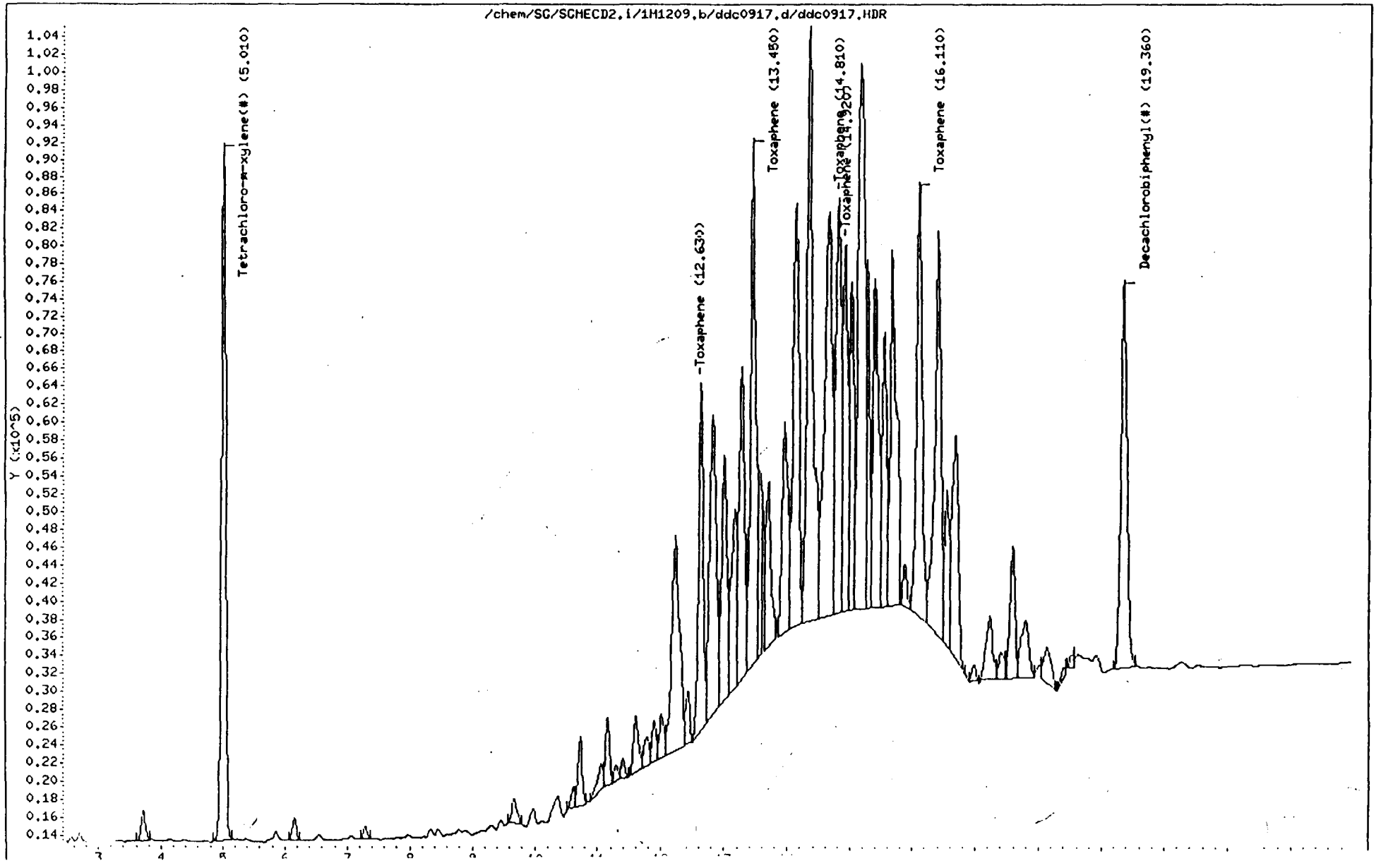
Column diameter: 0.53

Column phase: DB-5



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Operator: BW
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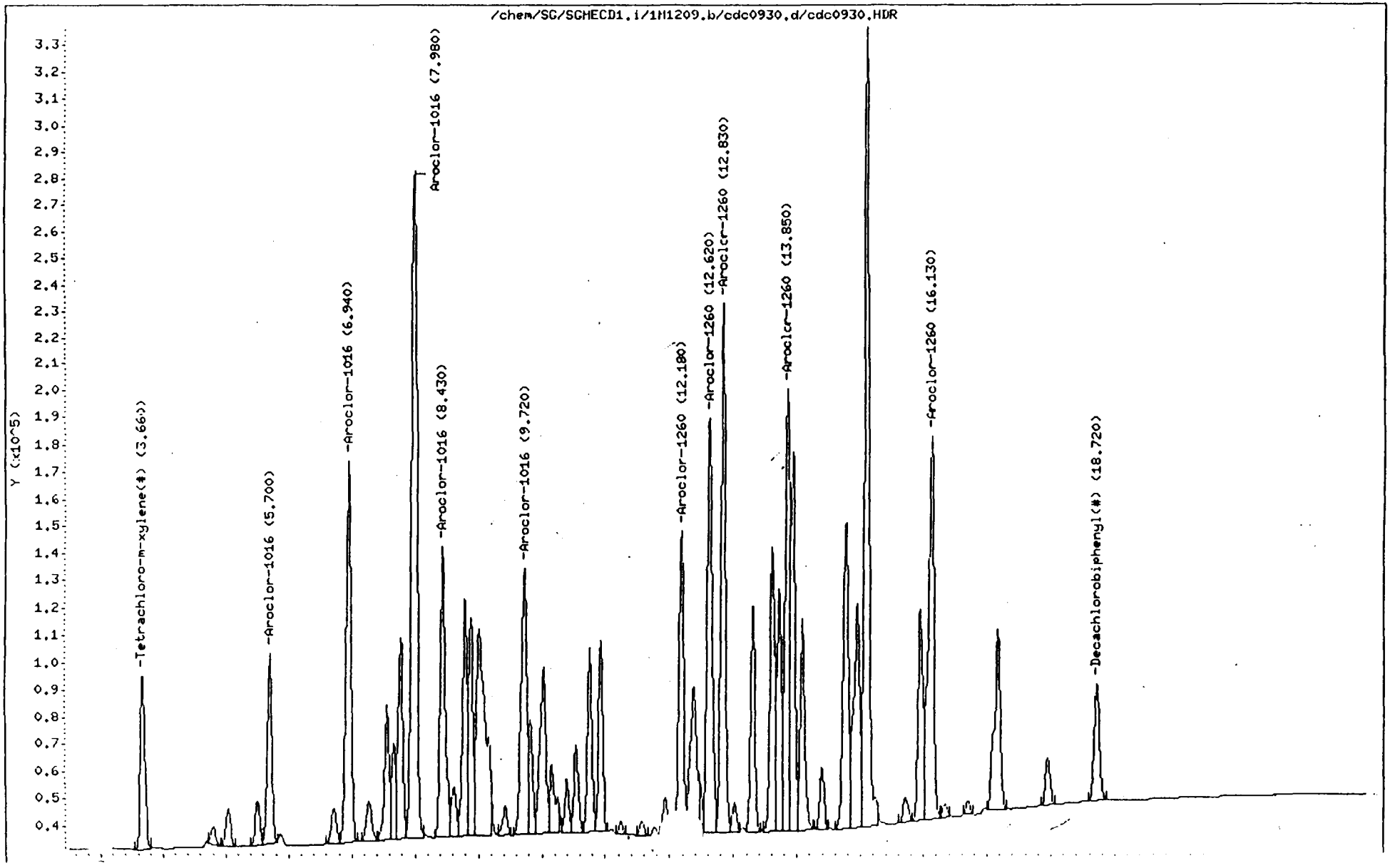
Column phase: DB-608



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Column phase: DB-5

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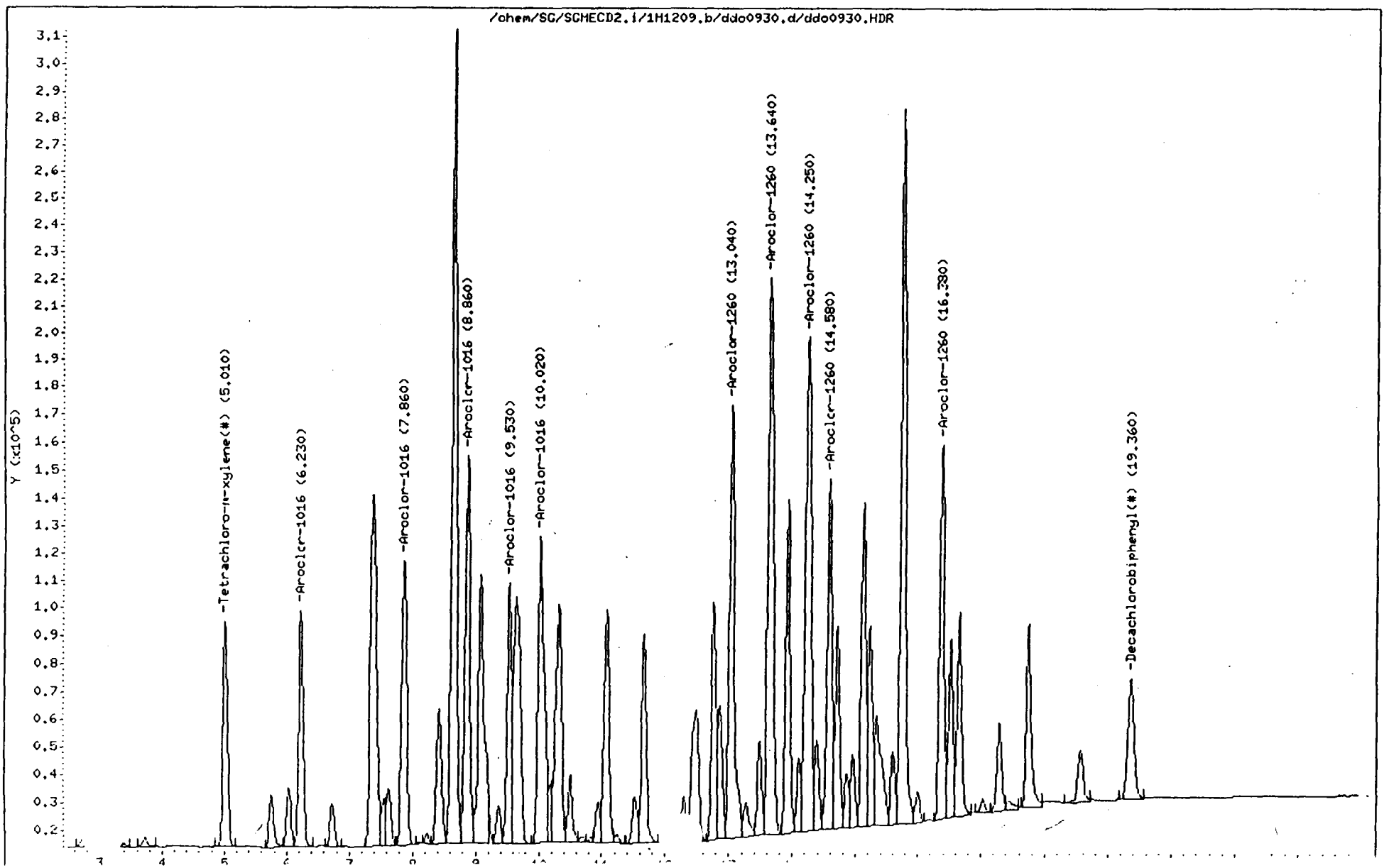
Column diameter: 0.53



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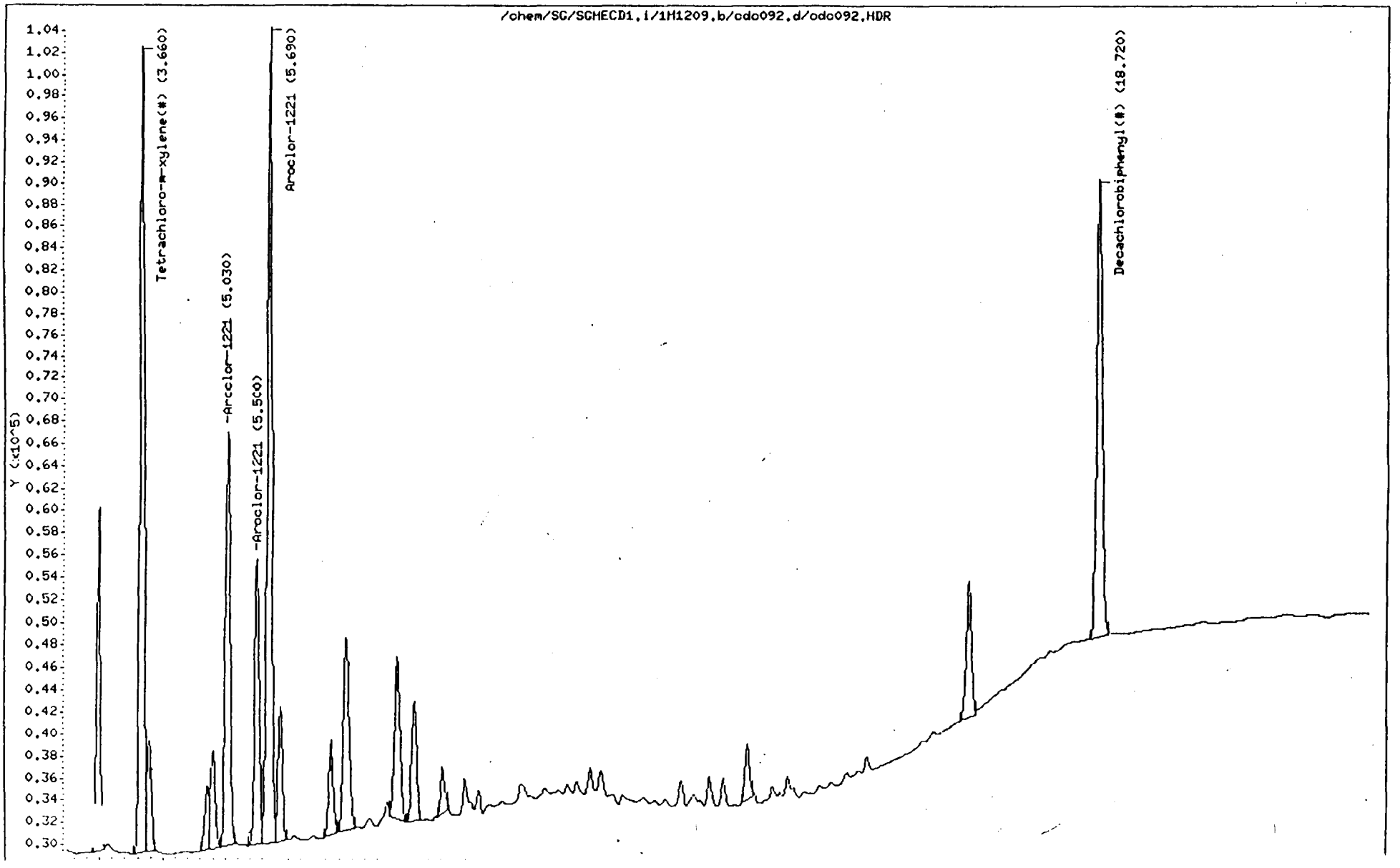
Operator: BW
Column diameter: 0.53



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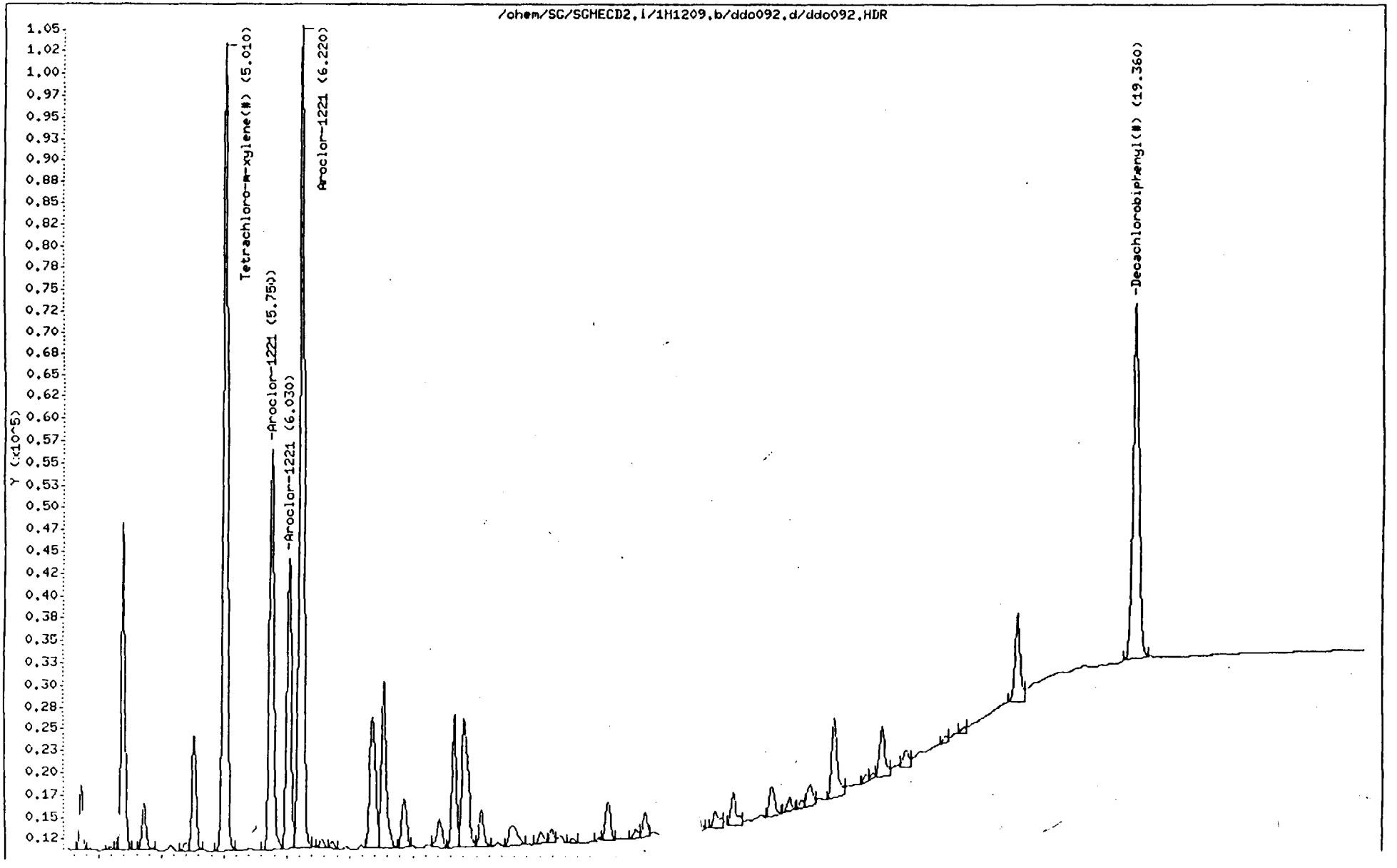
Sample Info: AR1221-04^1H1209

Column phase: DB-5

Instrument: SGHECD2.i

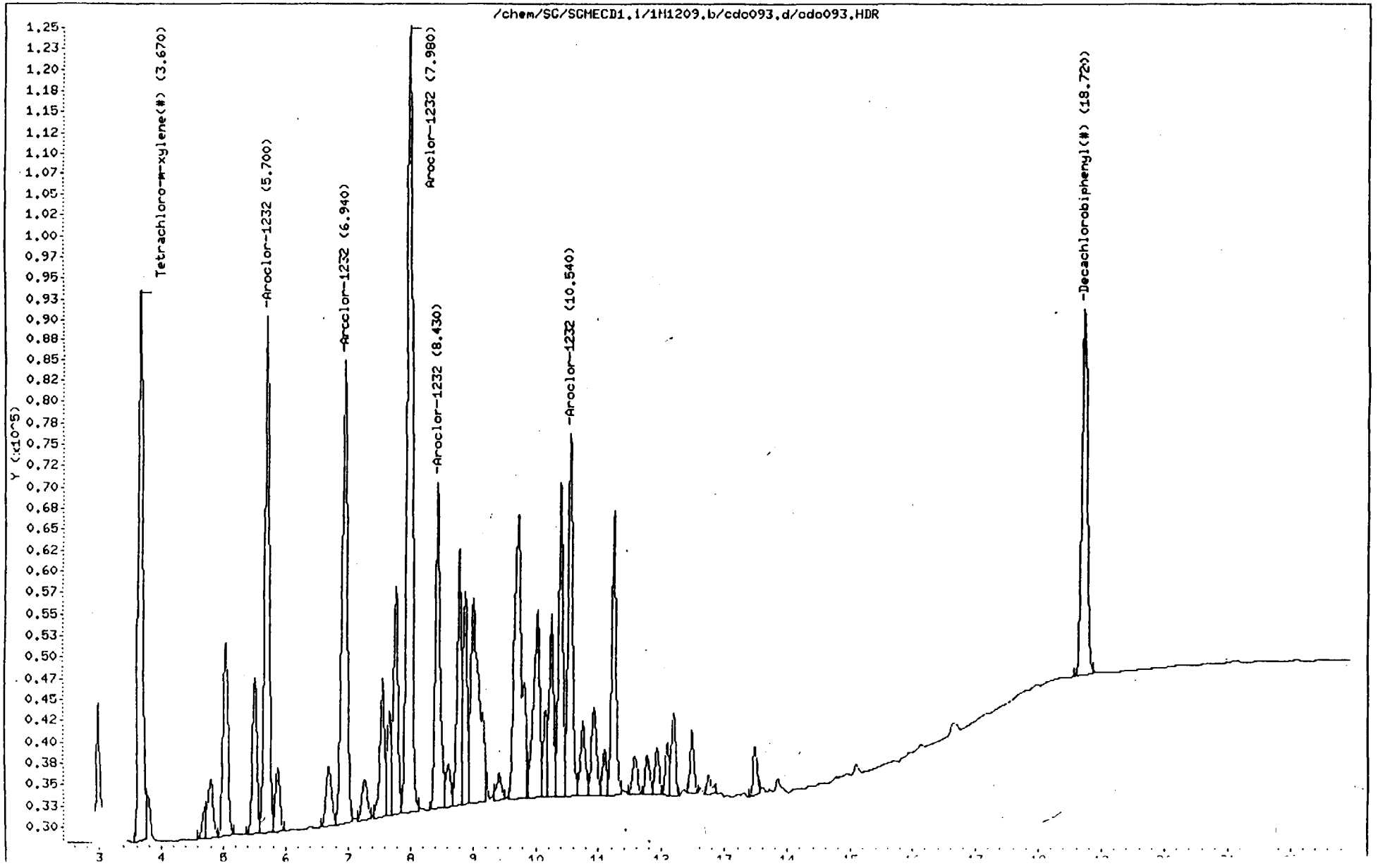
Operator: BW
Column diameter: 0.53



Data File: /chem/SC/SGMECD1.i/1M1209.b/cdc093.d
Date : 09-DEC-97 20:17:00
Client ID: AR1232-04
Sample Info: AR1232-04^1M1209

Instrument: SGMECD1.i
Operator: BW
Column diameter: 0.53

Column phase: DB-608

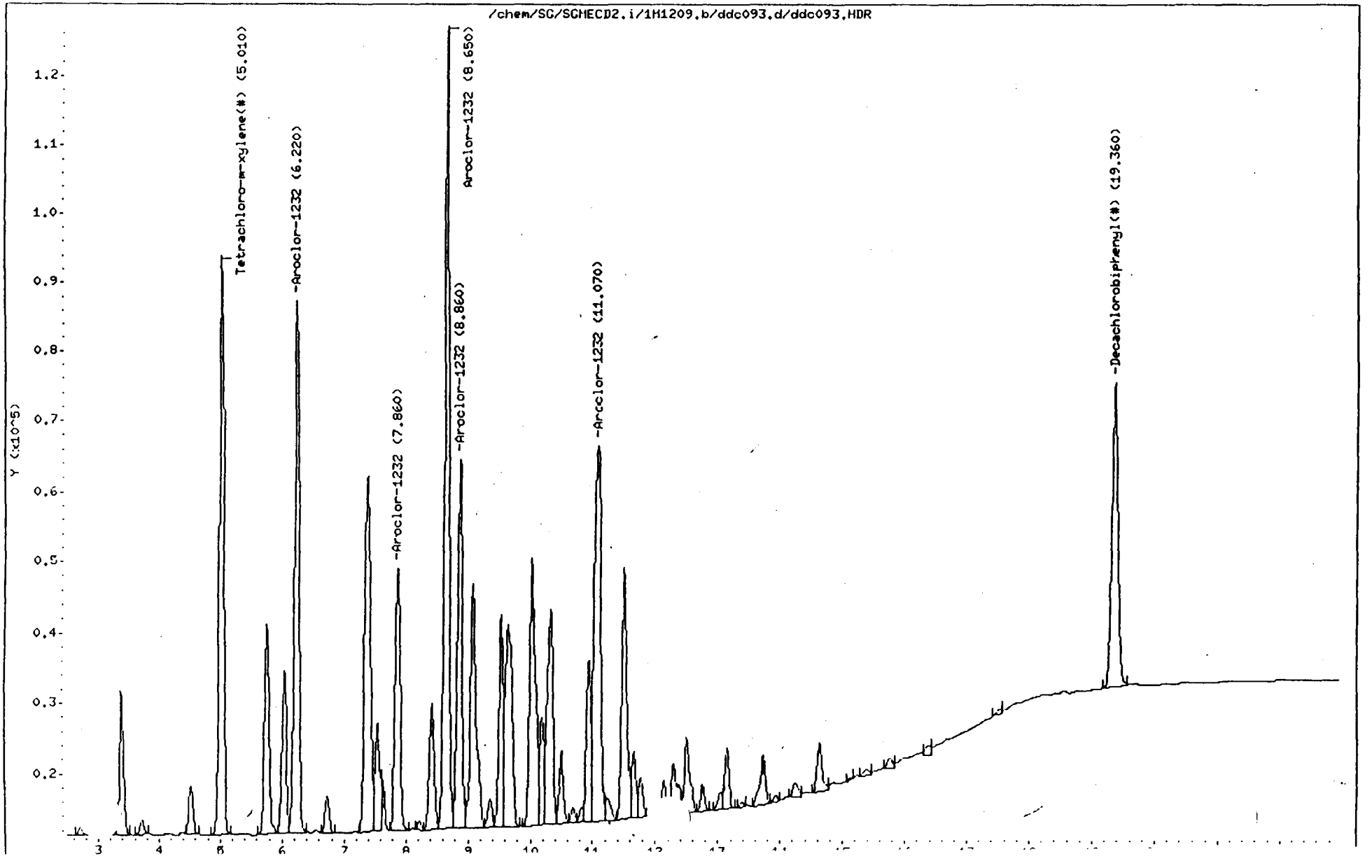


Data File: /chem/SG/SGHECD2.i/1H1209.b/ddc093.d
Date : 09-DEC-97 20:17:00
Client ID: AR1232-04
Sample Info: AR1232-04^1H1209

Column phase: DB-5

Instrument: SGHECD2.i

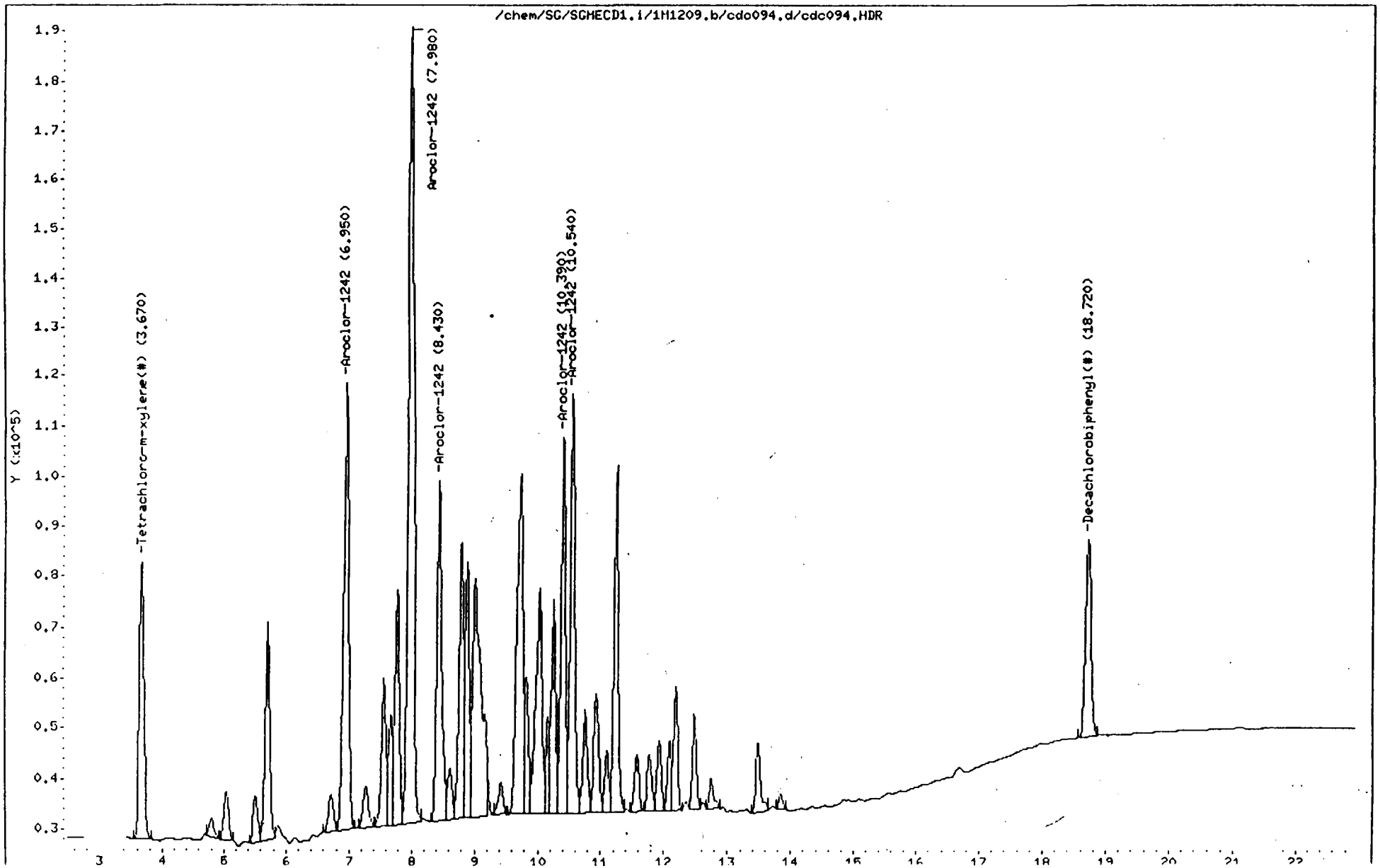
Operator: BW
Column diameter: 0.53



Data File: /chem/SG/SGHECD1.i/1H1209.b/cdc094.d
Date : 09-DEC-97 20:45:00
Client ID: AR1242-04
Sample Info: AR1242-04^1H1209

Instrument: SGHECD1.i
Operator: BW
Column diameter: 0.53

Column phase: DB-608



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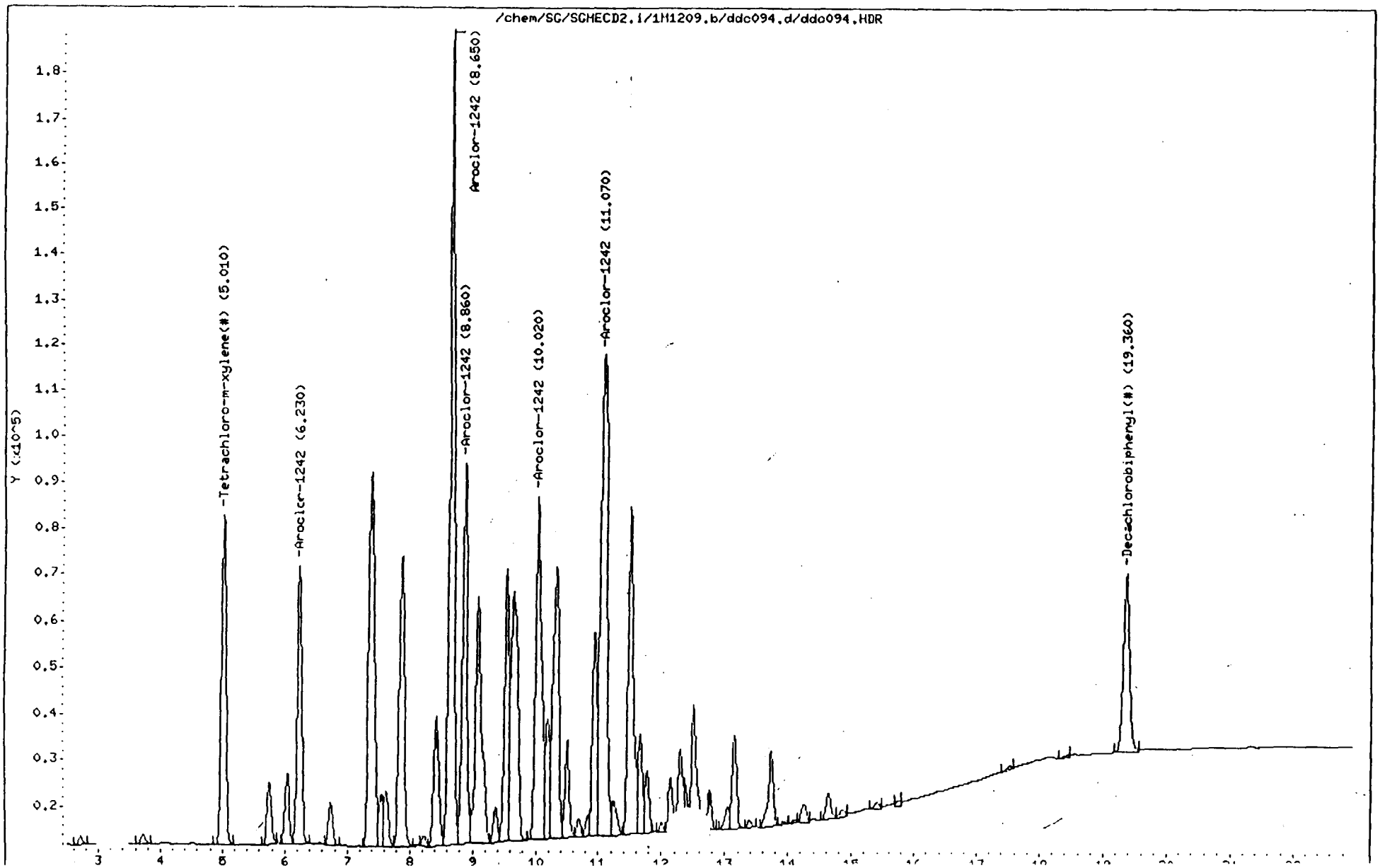
Date : 09-DEC-97 20:45:00
Client ID: AR1242-04
Sample Info: AR1242-04^1M1209

Column phase: DB-5

Instrument: SGHECD2.i

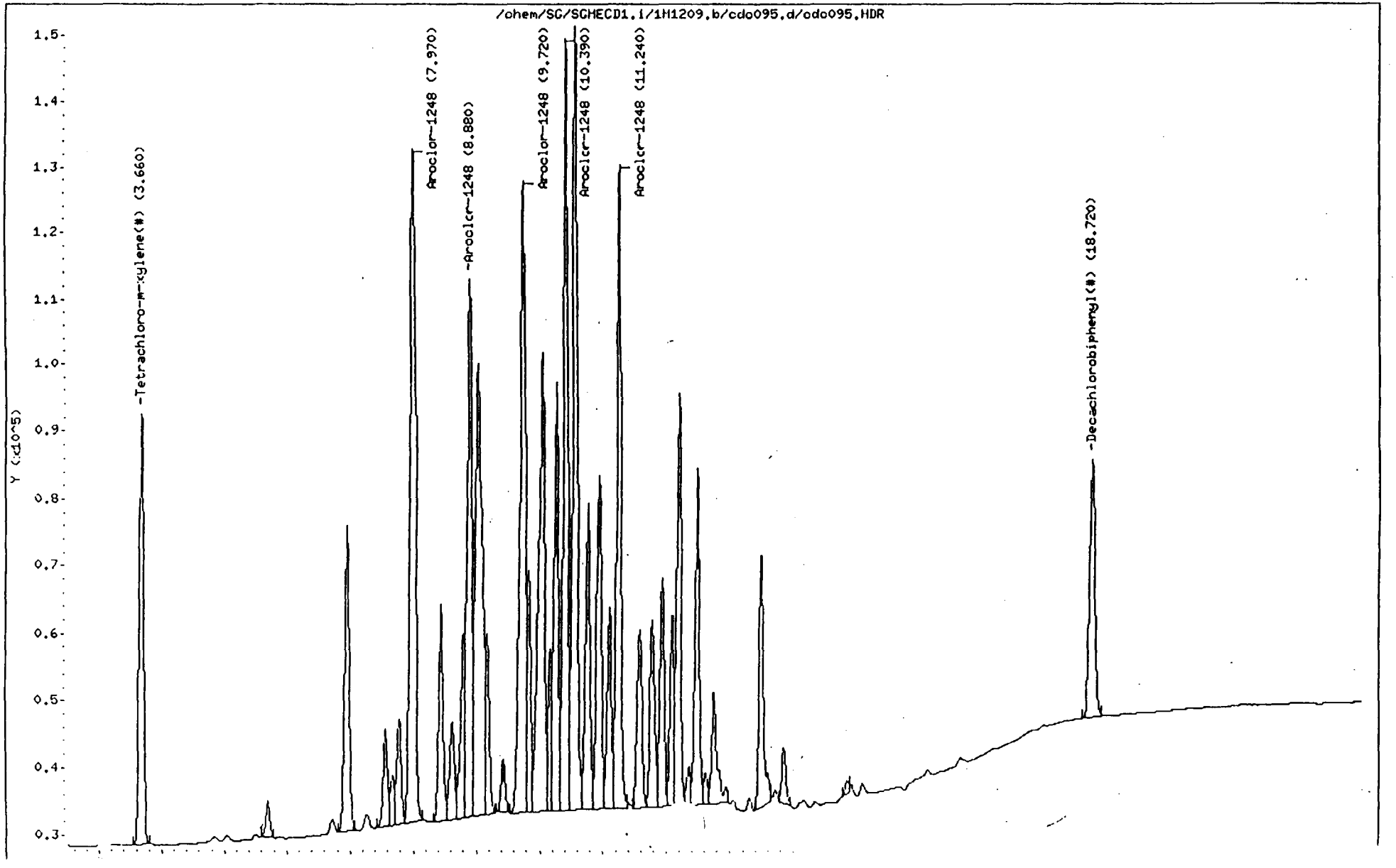
Operator: BW
Column diameter: 0.53



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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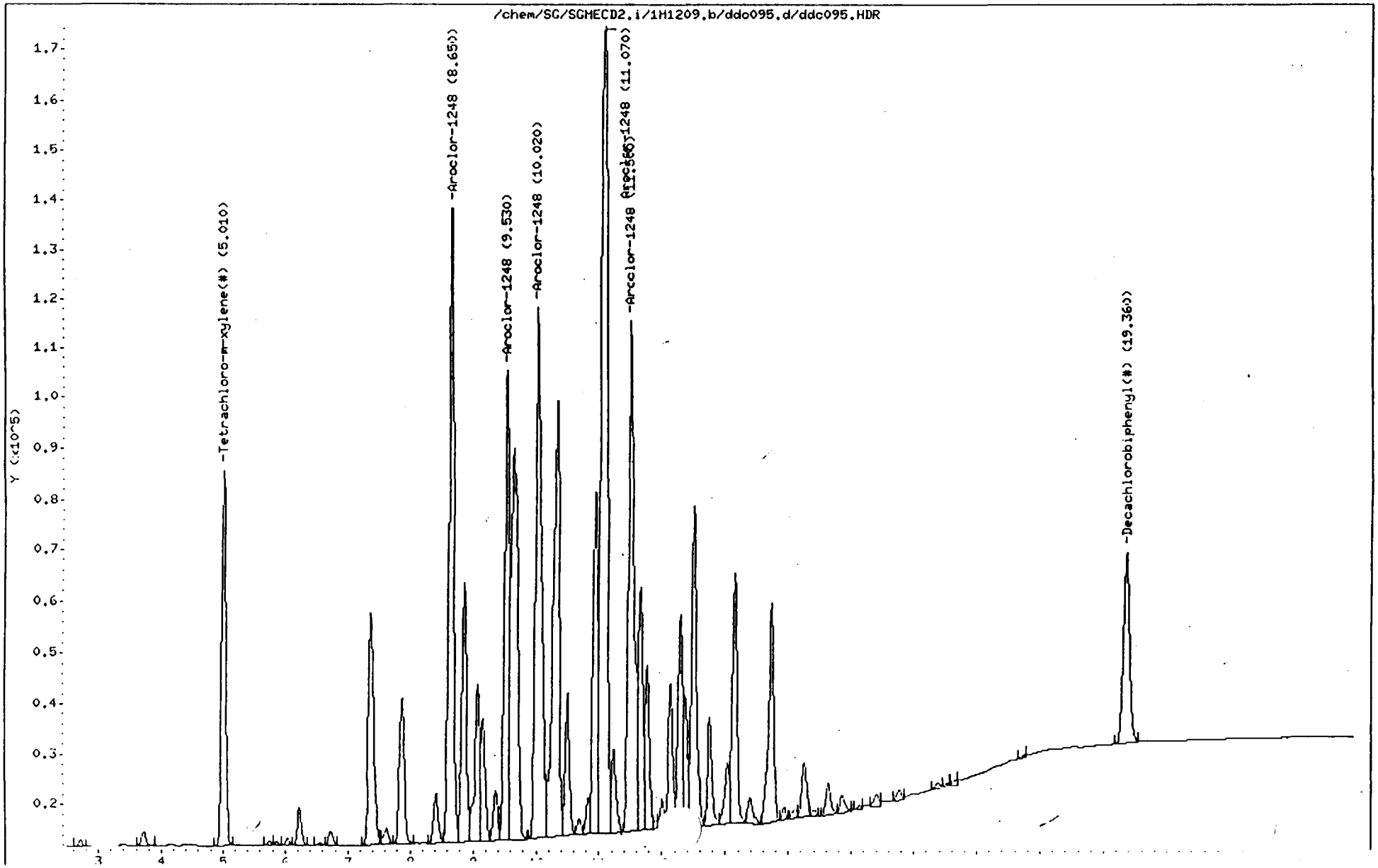
Date : 09-DEC-97 21:13:00
Client ID: AR1248-04
Sample Info: AR1248-04^1H1209

Instrument: SGHECD2.i

Operator: BW
Column diameter: 0.53

Column phase: DB-5

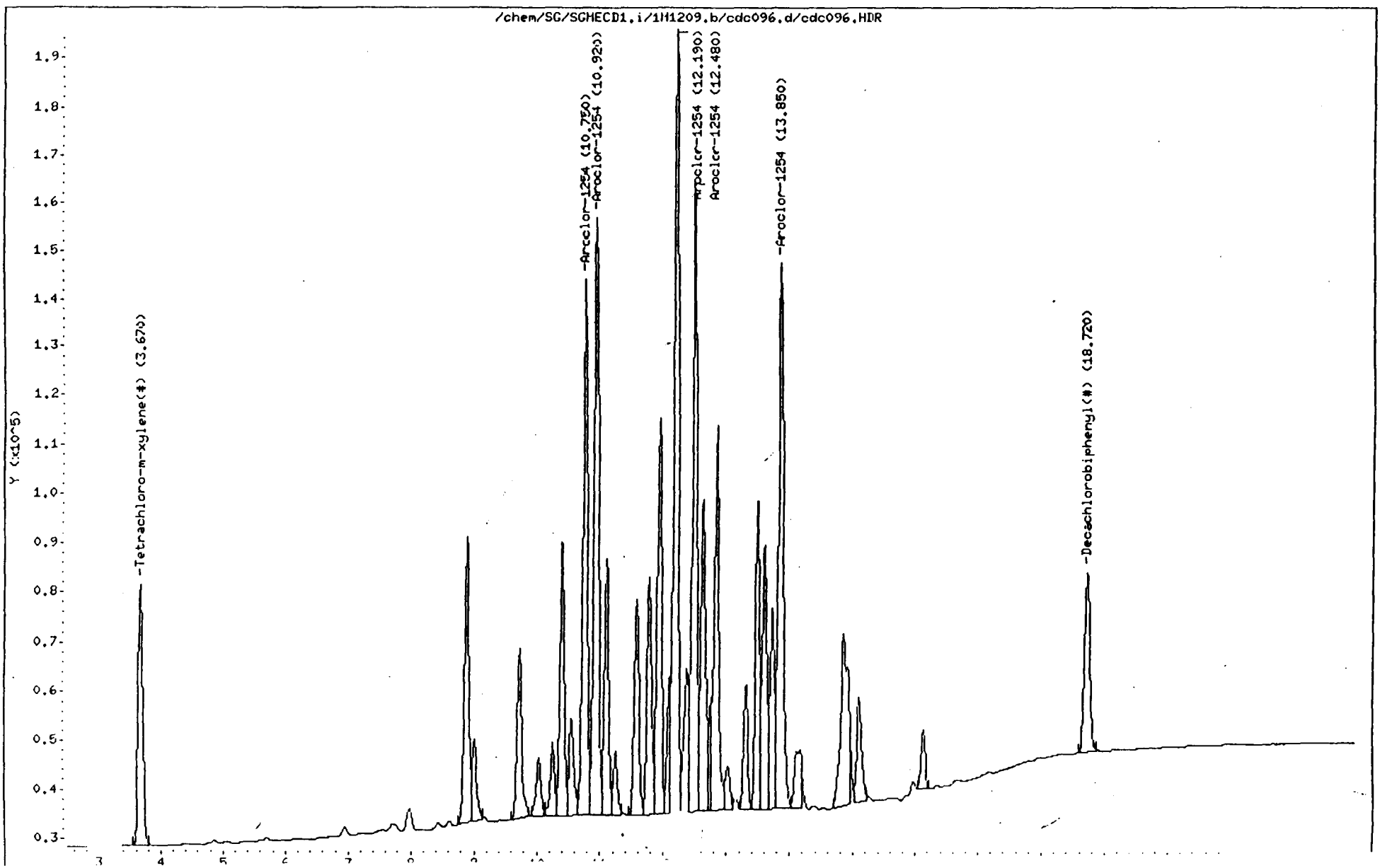


Data File: /chem/SG/SGHECD1.i/1M1209.b/cdc096.d
Date : 09-DEC-97 21:40:00
Client ID: AR1254-04
Sample Info: AR1254-04^1M1209

Instrument: SGHECD1.i

Operator: BW
Column diameter: 0.53

Column phase: DB-608



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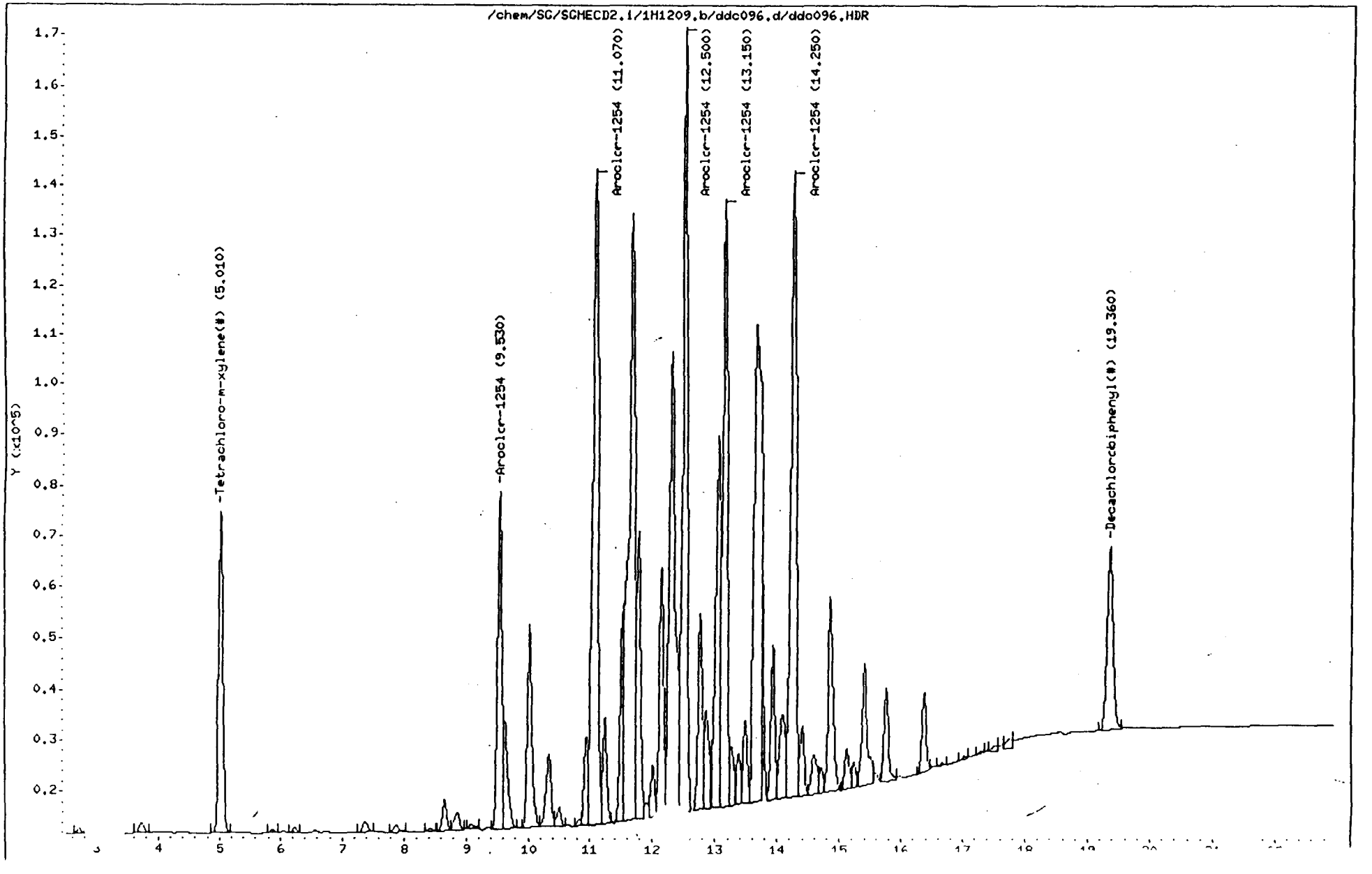
Date : 09-DEC-97 21:40:00
Client ID: AR1254-04
Sample Info: AR1254-04^1H1209

Instrument: SGHECD2.i

Operator: BW
Column diameter: 0.53

Column phase: DB-5

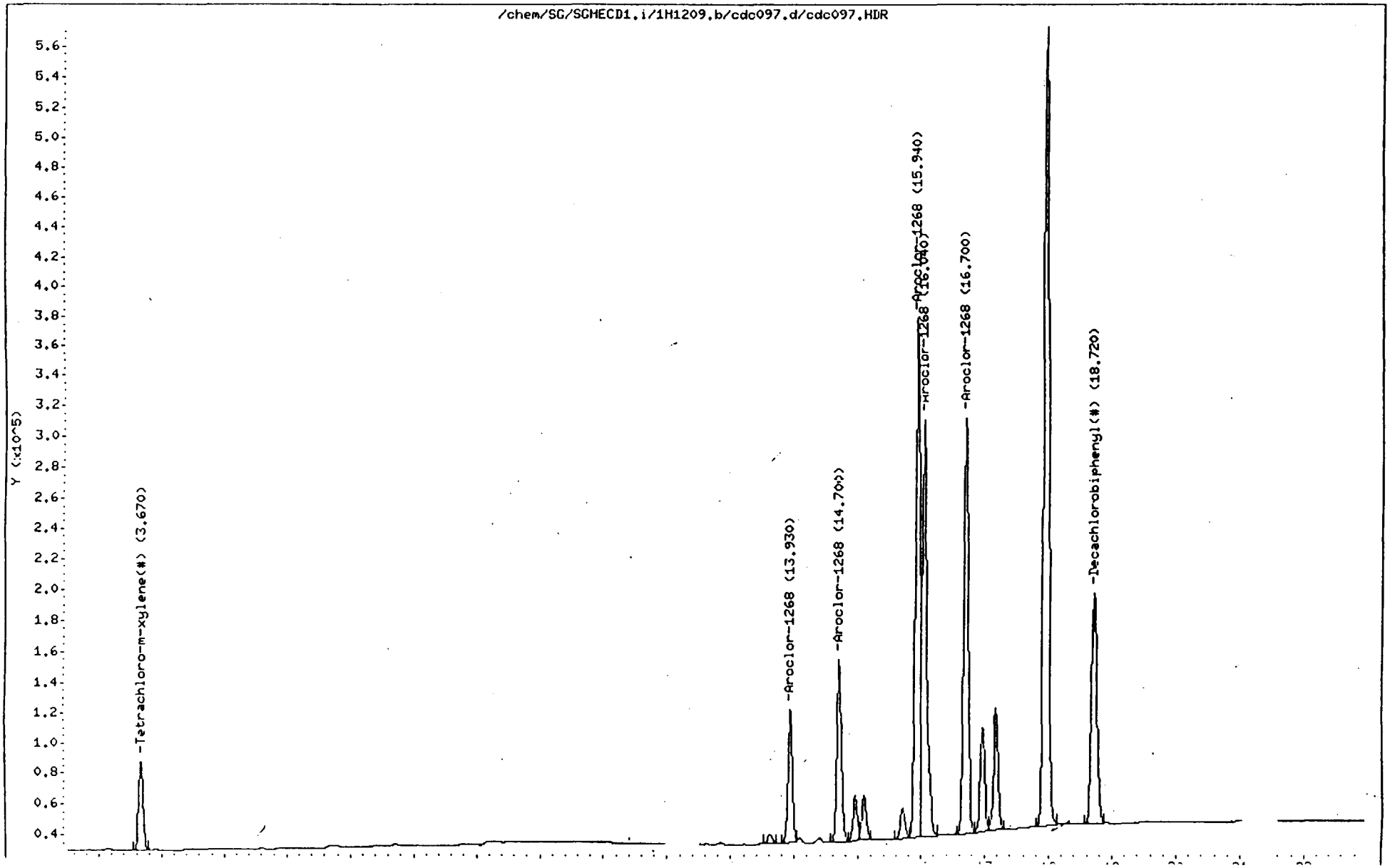


Data File: /chem/SG/SGHECD1.i/1H1209.b/cdc097.d
Date : 09-DEC-97 22:08:00
Client ID: AR1268-04
Sample Info: AR1268-04^1H1209

Instrument: SGHECD1.i

Operator: BW
Column diameter: 0.53

Column phase: DB-608

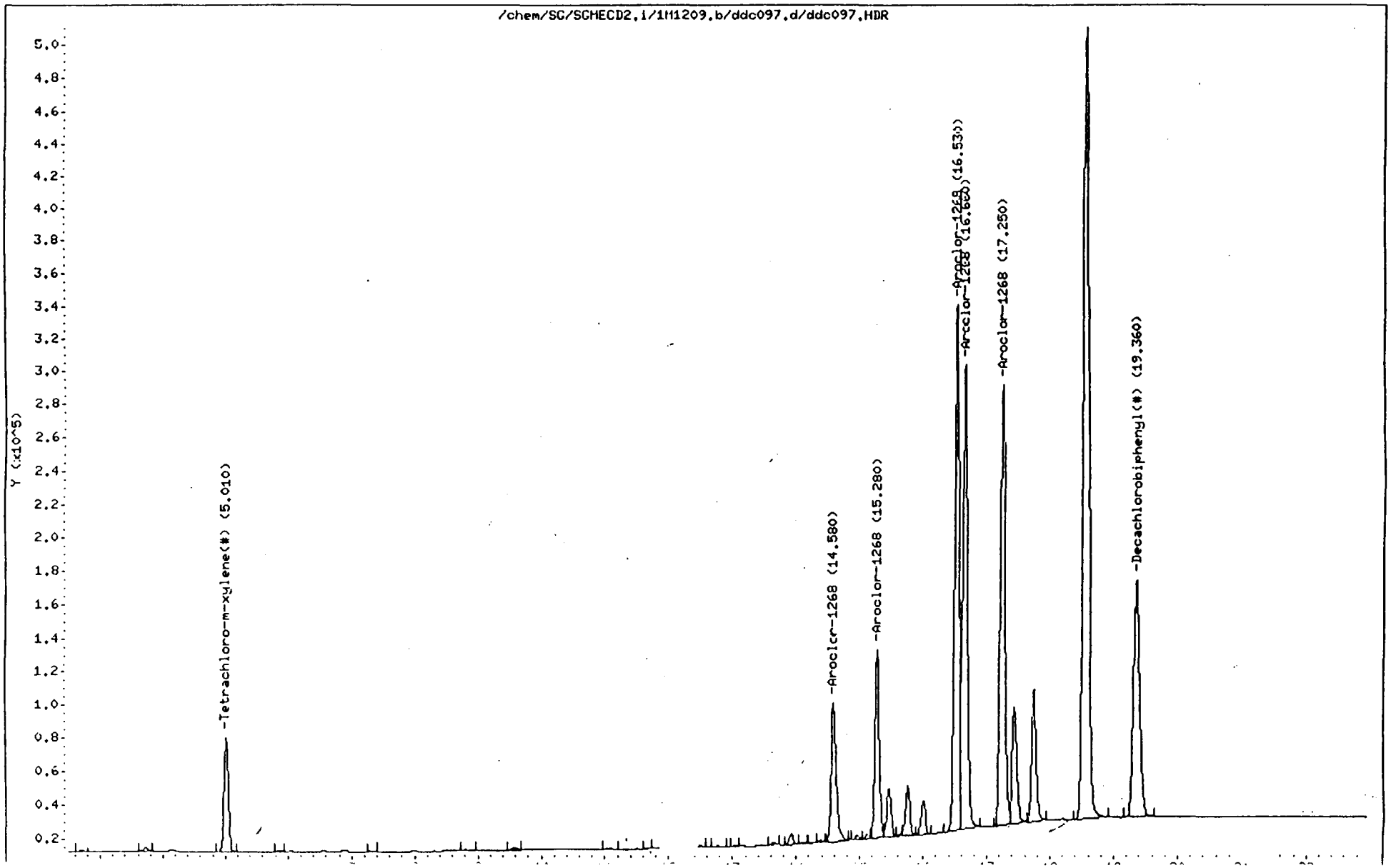


Data File: /chem/SC/SCHECD2.i/1H1209.b/ddc097.d
Date : 09-DEC-97 22:08:00
Client ID: AR1268-04
Sample Info: AR1268-04~1H1209

Instrument: SCHECD2.i

Operator: BW
Column diameter: 0.53

Column phase: DB-5



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: 10/22/97

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

Mode : 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
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	= 0.0222
#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 (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 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 (fL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	89 TO 111
QC SPIKE RATE	4
QC SPIKE VOLUME (fL)	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 ($NiNO_3$ 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 (fL) SOLUTION	BLANK	MODIFIER
BLANK	--	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 (fL)	15	
QC STANDARD CONCENTRATION	1.0	ug/l
QC STANDARD LIMITS (%)	90 TO 110	
QC SPIKE RATE	1	
QC SPIKE VOLUME (fL)	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 NIN03

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
LANK	--	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 # T-CAPE

Date Prepared 02.25.97

Analyst m m

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
STANDARD 3	50.0	3.6	0.222	0.216 0.228
QC				25Feb97 12:38
STANDARD 4	100.0	0.8	0.436	0.438 0.434
QC				25Feb97 12:42

(1)

Zeeman Se SW846/200 Series

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02-19

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 (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 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 (fL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	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 (fL) 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 (fL)	15
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	79 TO 121
QC SPIKE RATE	4
QC SPIKE VOLUME (fL)	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) Liquids 7060

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

OPERATOR MM
DATE 02/25/97
BATCH AS06 01286 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 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	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 (fL)

	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 (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	1.1 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 ($NiNO_3$ mod.) SW 846/200 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
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 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

2)

Zeeman As SW 846/200 series

AS05
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

02-24-97 LR

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)

ME ..OD 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	Sample Limits Table	: LCTAB
Autosampler Table	: AUTOTAB	Blank (AZ) Limits Table	: BLANKLCT
		QC Check Table	: QCTAB
		Recovery Check Table	: RQCTAB

OUTPUT 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 : As Se

Mode : Single Beam Absorption

I g Type : Peak Area Integ Time : 1.5 Sec. Delay Time : 1.2 Sec.
Graph 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
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	= 0.0123
#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.
1:	BlankSTD	= 0.00	= 0.0000
#2:	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 (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

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 (%)	0 TO 200
QC SPIKE RATE	4
QC SPIKE VOLUME (fL)	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) SB846

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

Sool
02-12-9
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 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 (fL)	20
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	39 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	1.3 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
STANDARD 4	100.0	0.5	0.364	0.365	0.363
QC				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
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 # LT-2
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 T1 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 (fL) 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 (fL)	15
QC STANDARD CONCENTRATION	50.00 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	1
QC SPIKE VOLUME (fL)	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 SM846/200 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
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

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
QC				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 (fL) 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 (fL)	10
QC STANDARD CONCENTRATION	50.0 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	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
ANK	--	20	3
TANDARD 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 GF = 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 LC

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	0.0		0.034	0.042 0.025
QC				14Feb97 13:03
				CONCENTRATION LESS THAN IDL
BLANK	0.0		-0.020	-0.019 -0.021
QC				14Feb97 13:09
				CONCENTRATION LESS THAN IDL
STANDARD 1	5.0	0.3	0.052	0.052 0.052
QC				14Feb97 13:13
STANDARD 2	10.0	2.2	0.103	0.104 0.101
QC				14Feb97 13:18
STANDARD 3	50.0	3.2	0.498	0.486 0.509
				14Feb97 13:22
STANDARD 4	100.0	0.3	0.924	0.922 0.926
QC				14Feb97 13:26

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

Zeeman Li "SW846-like"

OPERATOR LR
DATE 02/27/97
BATCH LI05 70676 AUDIT,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 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 (fL)

	SOLUTION	BLANK	MODIFIER
BLANK	--	8	12
STANDARD 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 (fL)	8
QC STANDARD CONCENTRATION	25.00 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	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 QC	0.00		0.002	0.001 0.002 27Feb97 06:53 CONCENTRATION LESS THAN IDL
STANDARD 1 QC	5.00	10.1	0.007	0.007 0.006 27Feb97 06:57
STANDARD 2 QC	10.00	3.6	0.017	0.017 0.018 27Feb97 07:00
STANDARD 3 QC	20.00	0.9	0.037	0.037 0.036 27Feb97 07:05
STANDARD 4 QC	50.00	1.8	0.097	0.095 0.098 27Feb97 07:08

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 (fL)

	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 (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	0.3 ug/L
REQUIRED DETECTION LIMIT	10.0 ug/L

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

TJA Video 22 Ni SW846/200 series

Atomizer: Furnace
Matrix: 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

OUTPUT 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

mm/LL

Varian SpectrAA 300/400 Zeeman
QC Protocol Report

Zeeman Cd SW846/200 series

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

SAMPLER PARAMETERS

VOLUMES (fL)

	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 (fL)	15
QC STANDARD CONCENTRATION	5.00 ug/L
QC STANDARD LIMITS (%)	40 TO 160
QC SPIKE RATE	0
QC SPIKE VOLUME (fL)	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

Zeeman Ag SW846/200 series

OPERATOR LR
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 **

Output 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.
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 **

Output Mode: Concentration
erride Print Limits? Yes
verride 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.
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

TJA Video 22 Pb/Ti SW846, 200 series

Atomizer: Furnace
rix: 1 % HNO3 IN DI WATER

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

PUT 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

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.

2.2 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

CAL 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

CAL TANDARD	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

*RL 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

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 : 250-260°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

10.2 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

10.2.1.2 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.

10.2.1.4 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:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_1} (RRF_i - RRF_{avg})^2}{n-1}}$$

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%

10.2.9 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

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

.1.1.1 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.

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

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.

1.3.1 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.

2.2 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 Target Version : 3.12
Last Update : 04-Dec-96 14:43:5 Number of Cpnds : 91
Data Type : MS DATA

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 mf 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
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

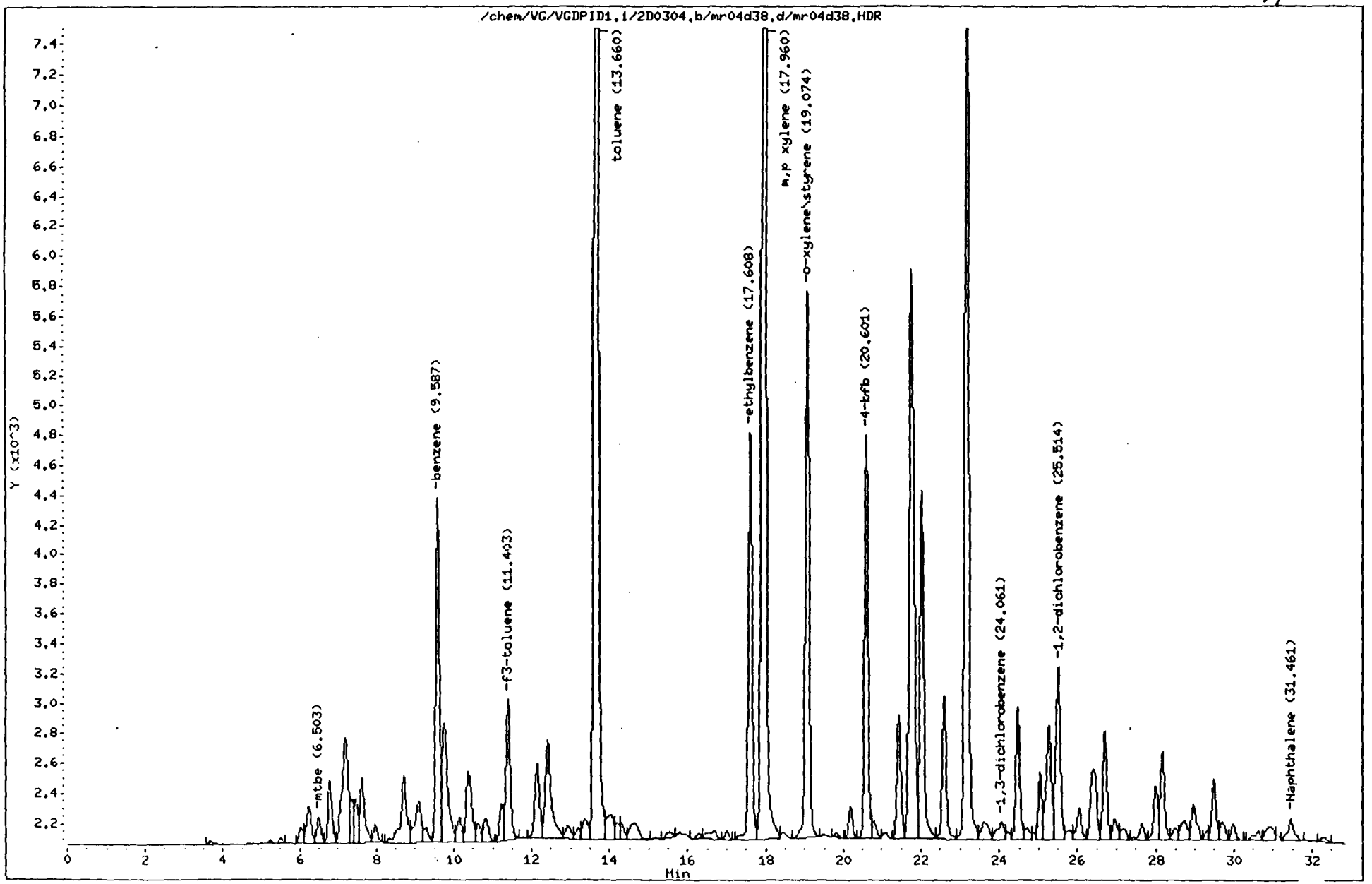
Appendix C
VG15 EXAMPLE CHROMATOGRAMS

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



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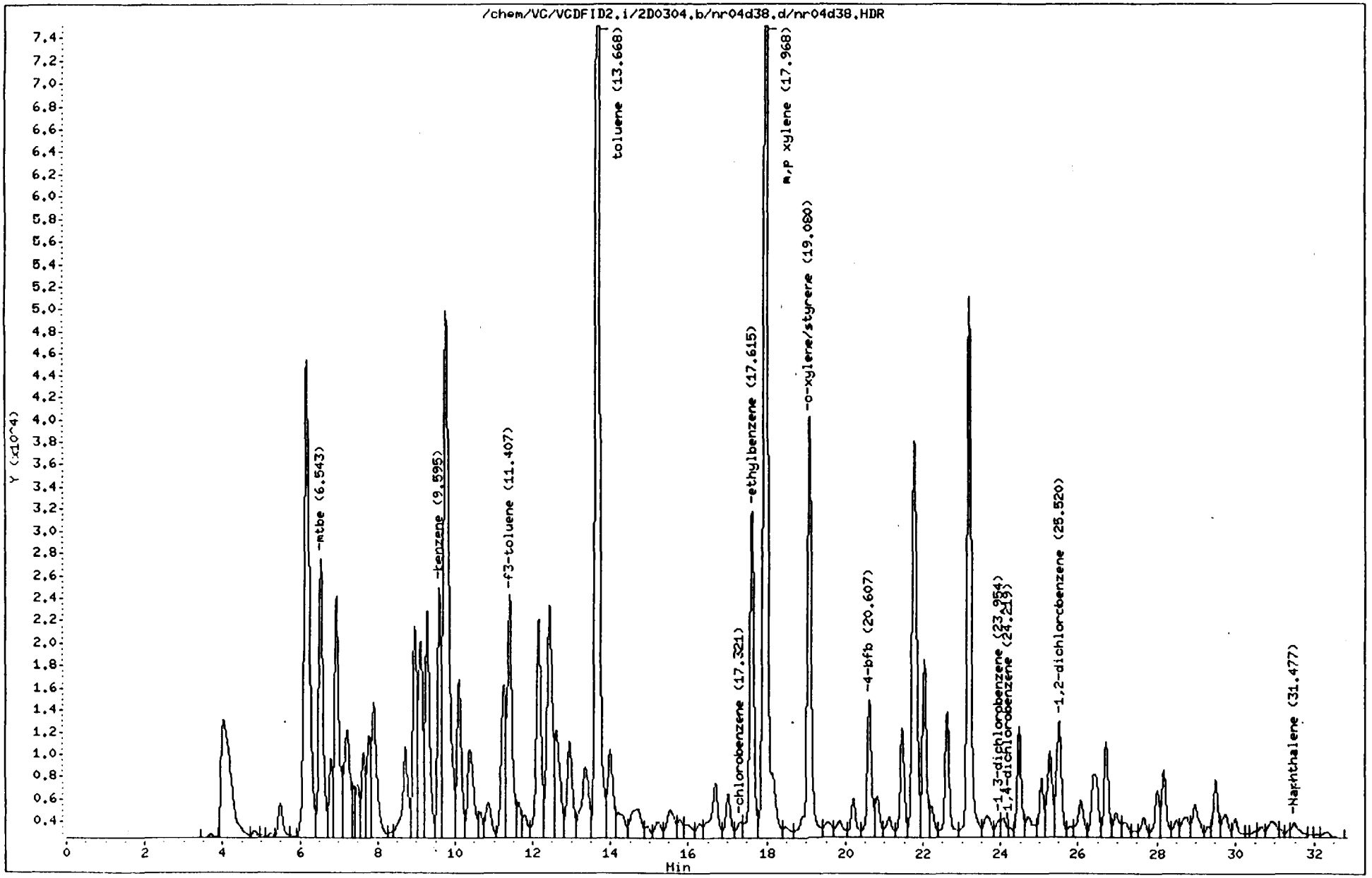
Date : 06-MAR-97 08:52:00
Client ID: VGASOL 1.5
Sample Info: VGASOL 1.5~2D0304

Instrument: VG.i
Operator: VGD
Column diameter: 0.53

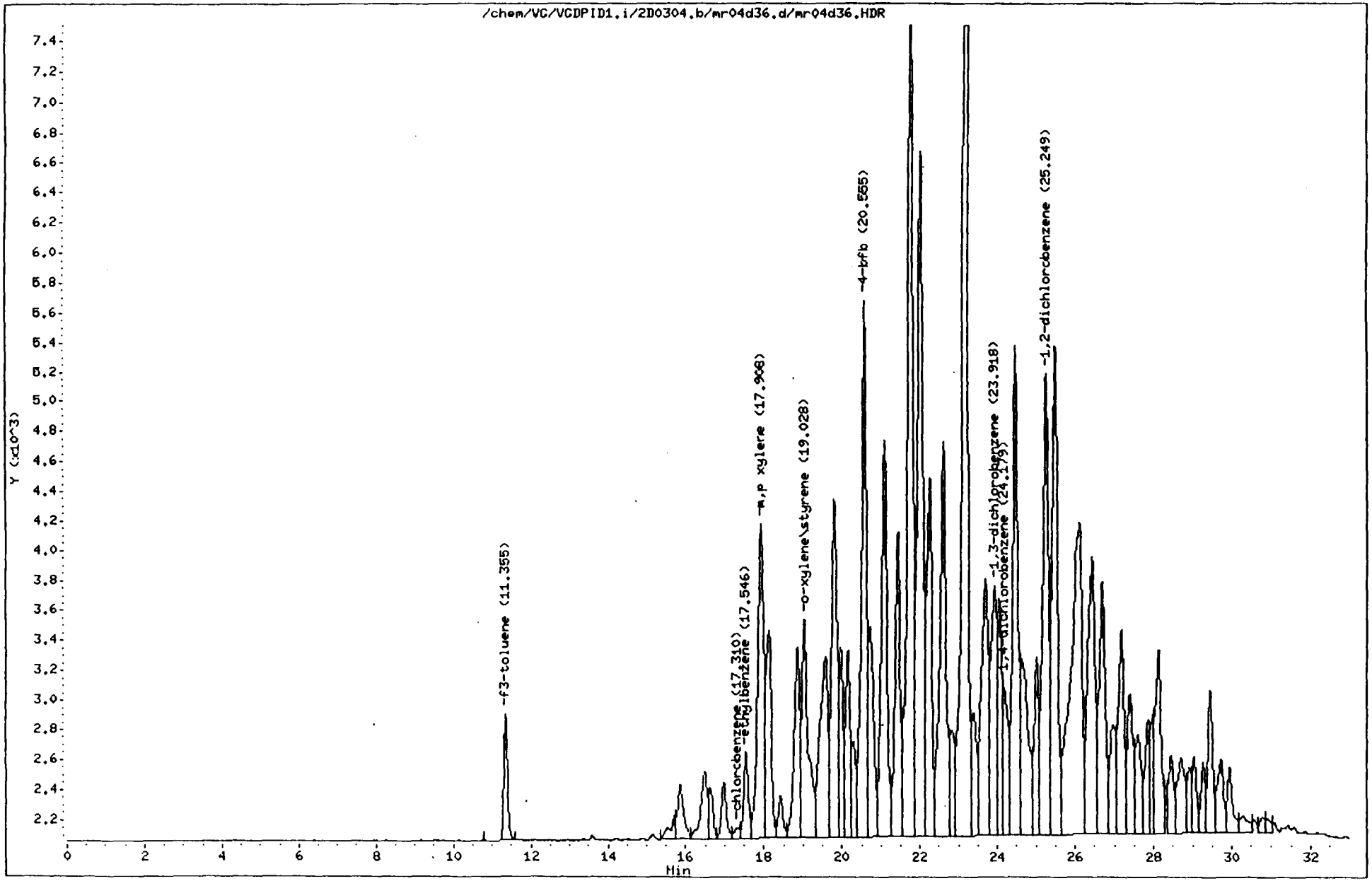
Column phase: DB624

B



Data File: /chem/VG/VGDPID1.i/2D0304.b/mr04d36.d
Date : 06-MAR-97 07:18:00
Client ID:
Instrument: VGDPID1.i
Sample Info: VMINER-900~2D0304
Purge Volume: 25.0
Operator: VGD
Column phase: DB624
Column diameter: 0.53

C



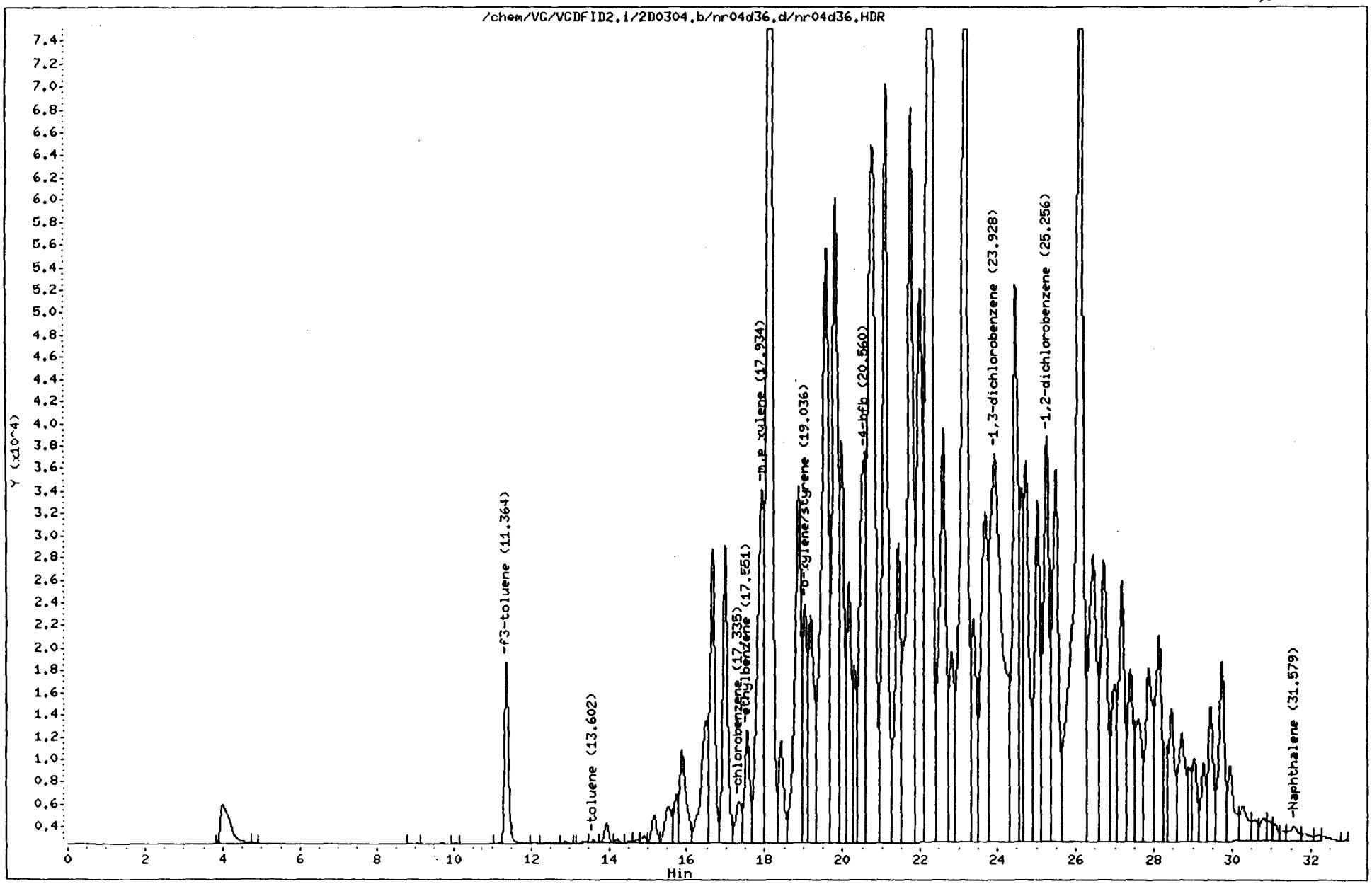
Data File: /chem/VG/VGDFID2.i/2D0304.b/nr04d36.d
Date : 06-MAR-97 07:18:00
Client ID: MINERAL SPIRITS
Sample Info: VHINER-900~2D0304

Instrument: VG.i

Operator: VGD
Column diameter: 0.53

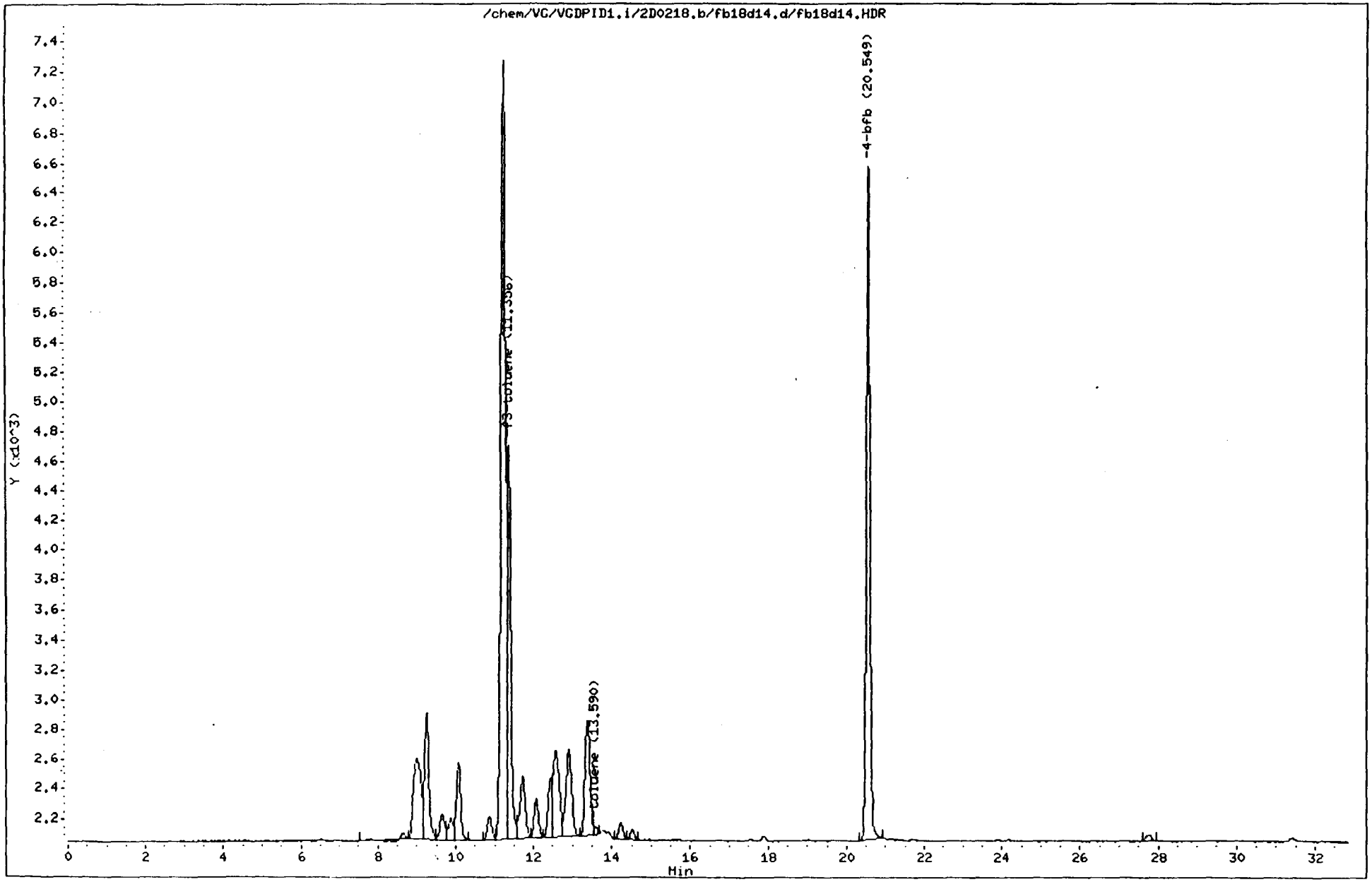
Column phase: DB624

D



Data File: /chem/VG/VGDPID1.i/2D0218.b/fb18d14.d
Date : 18-FEB-97 21:05:00
Client ID: VGLAC-800
Instrument: VGDPID1.i
Sample Info: VGLAC-800~2D0218
Purge Volume: 25.0
Operator: VGD
Column phase: DB624
Column diameter: 0.53

E



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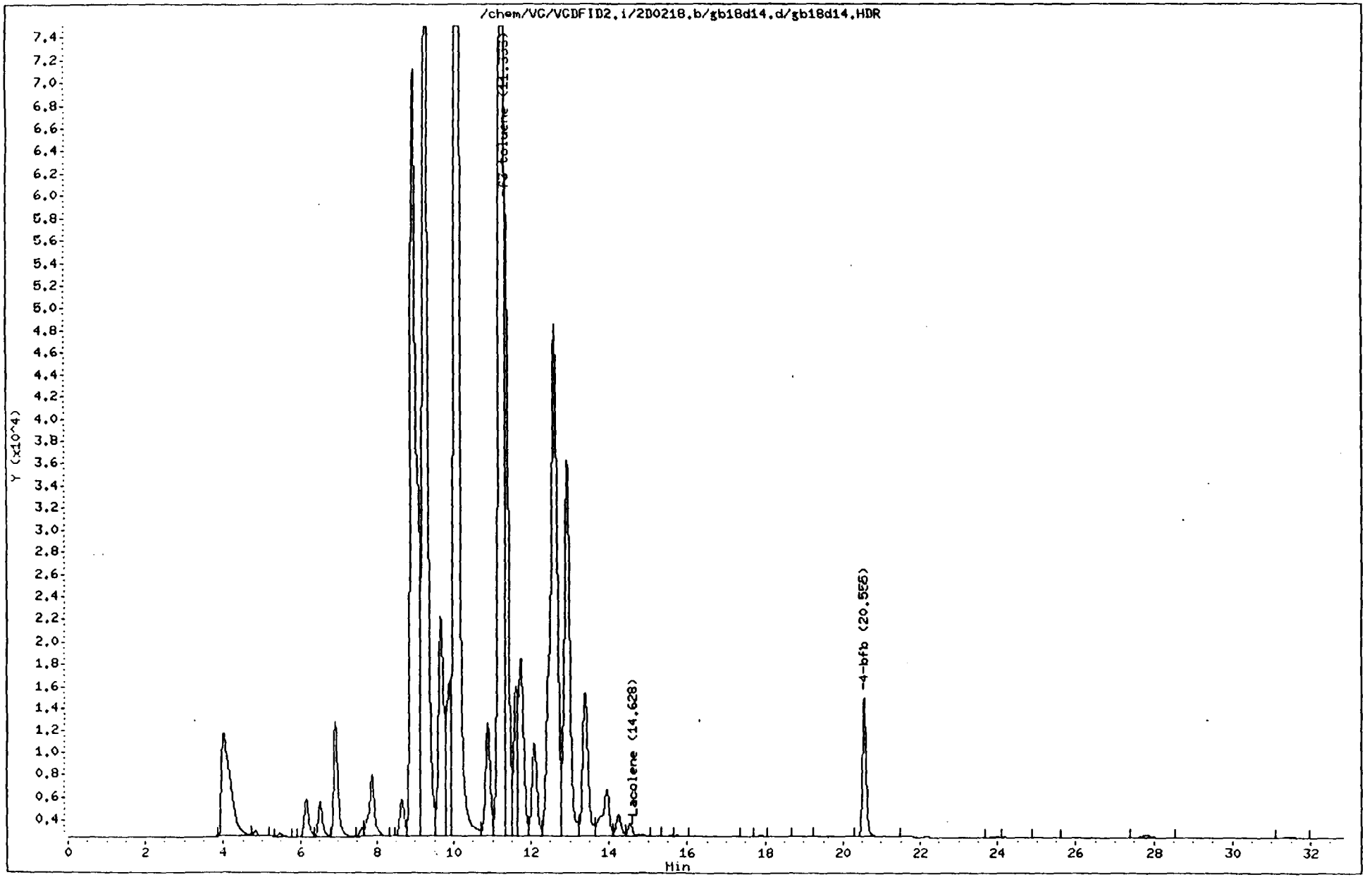
Date : 18-FEB-97 21:05:00
Client ID: VGLAC-800
Sample Info: VGLAC-800^2D0218
Purge Volume: 25.0
Column phase: DB624

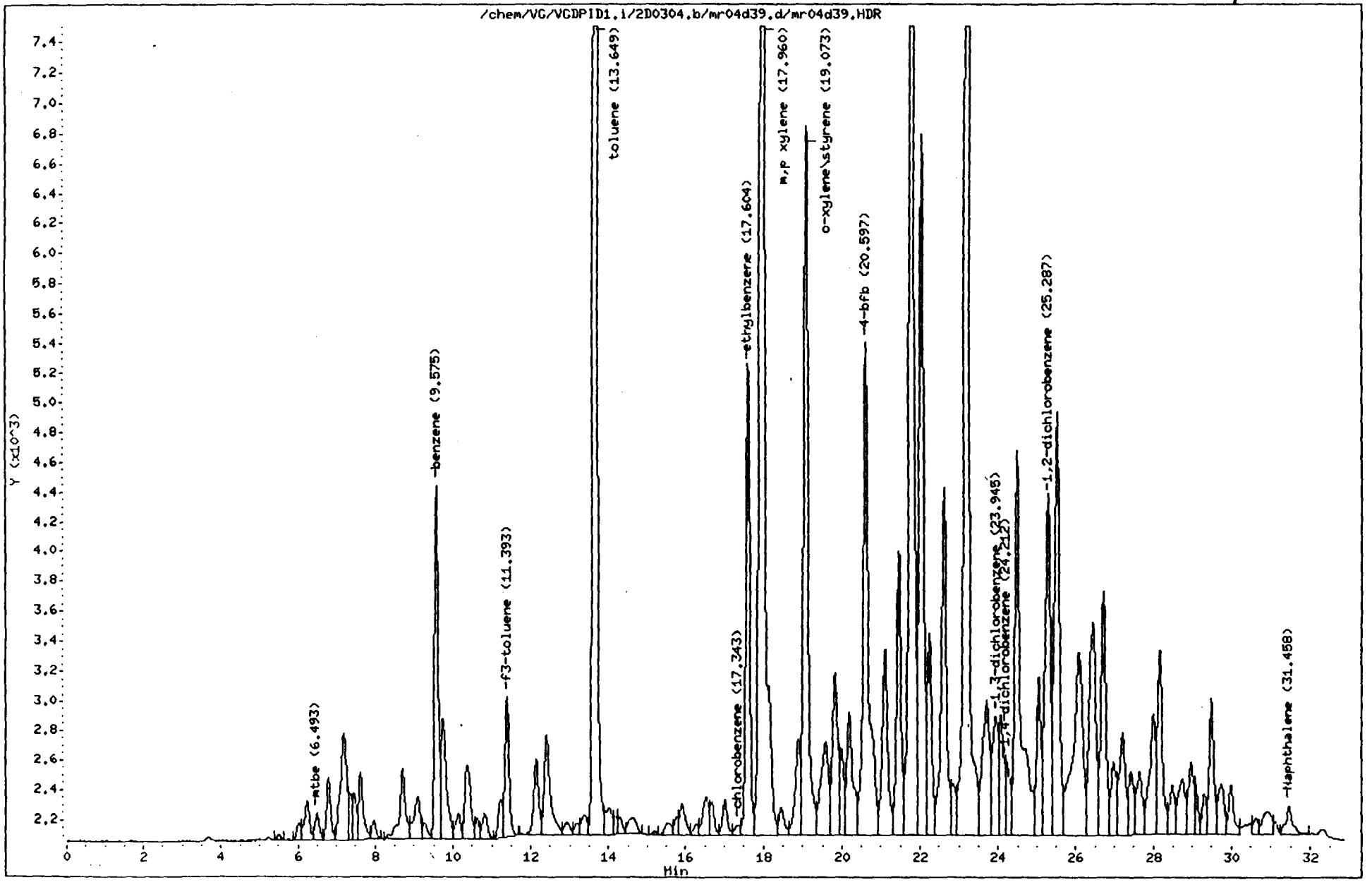
Instrument: VGDFID2.i
Operator: VGD
Column diameter: 0.53

F



Data File: /chem/VG/VGDPID1.i/2D0304.b/mr04d39.d
Date : 06-MAR-97 09:38:00
Client ID: VHI/GS
Instrument: VGDPID1.i
Sample Info: VHI/GS ~2D0304
Purge Volume: 25.0
Operator: VGD
Column phase: DB624
Column diameter: 0.53

G



Data File: /chem/VG/VGDFID2.i/2D0304.b/nr04d39.d

Page 1

Date : 06-MAR-97 09:38:00

Client ID: VMI/GS

Instrument: VG.i

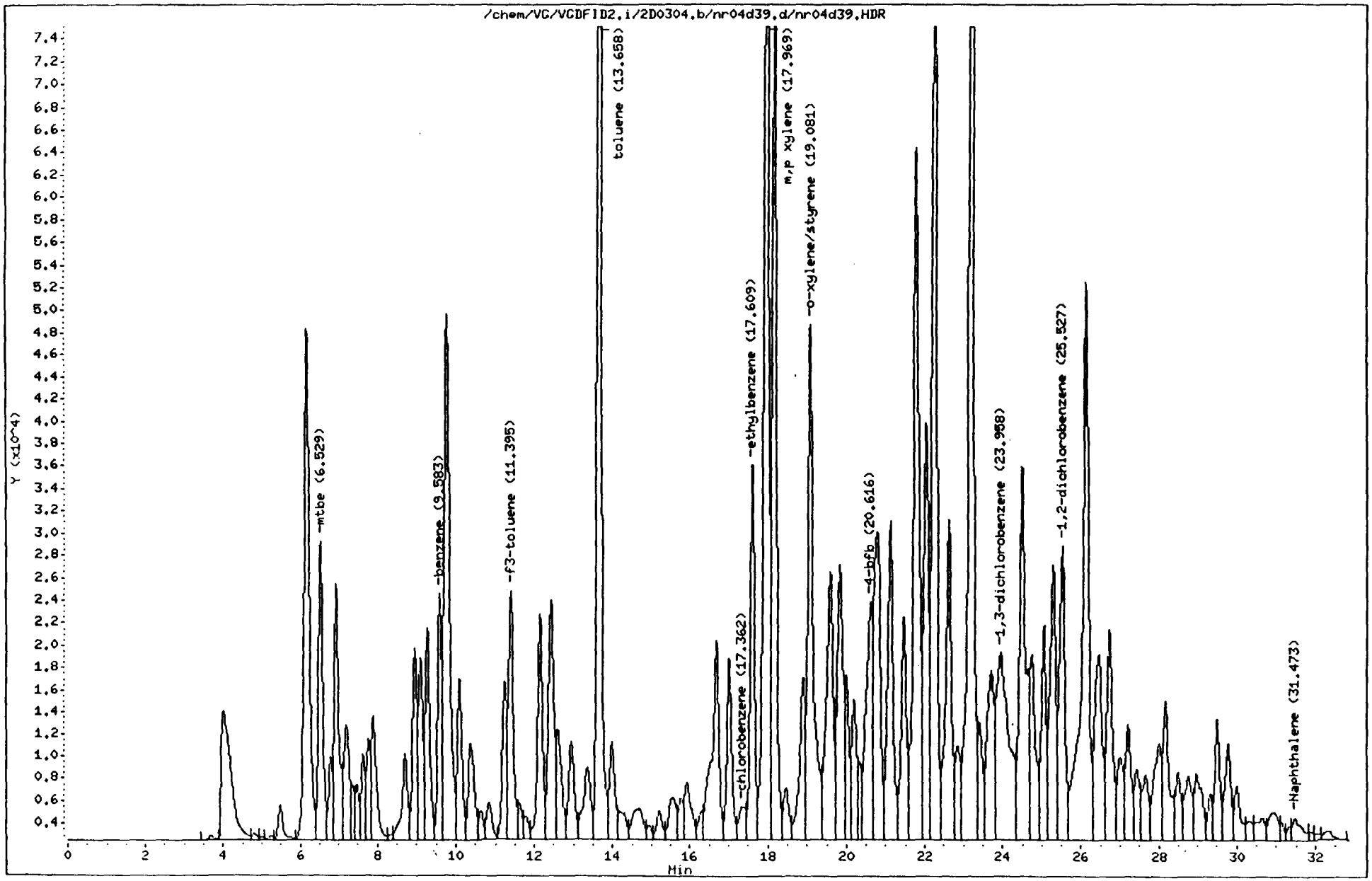
Sample Info: VMI/GS ~2D0304

Operator: VGD

Column phase: DB624

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 rrange 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.

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	81.00
	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 PENTACHLOROEHTANE	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

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 + \ldots RFn}{n}$$

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

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

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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

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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.

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 by 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 terms 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.

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}}$$

where
RF_i = response factor of the individual calibration level
$\overline{RF}$ = average response factor

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:

$$\%Difference = \frac{RF_{init} - RF_{cont}}{RF_{init}} \times 100$$

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:

$$\%Drift = \frac{result - expected}{expected} \times 100$$

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

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

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.

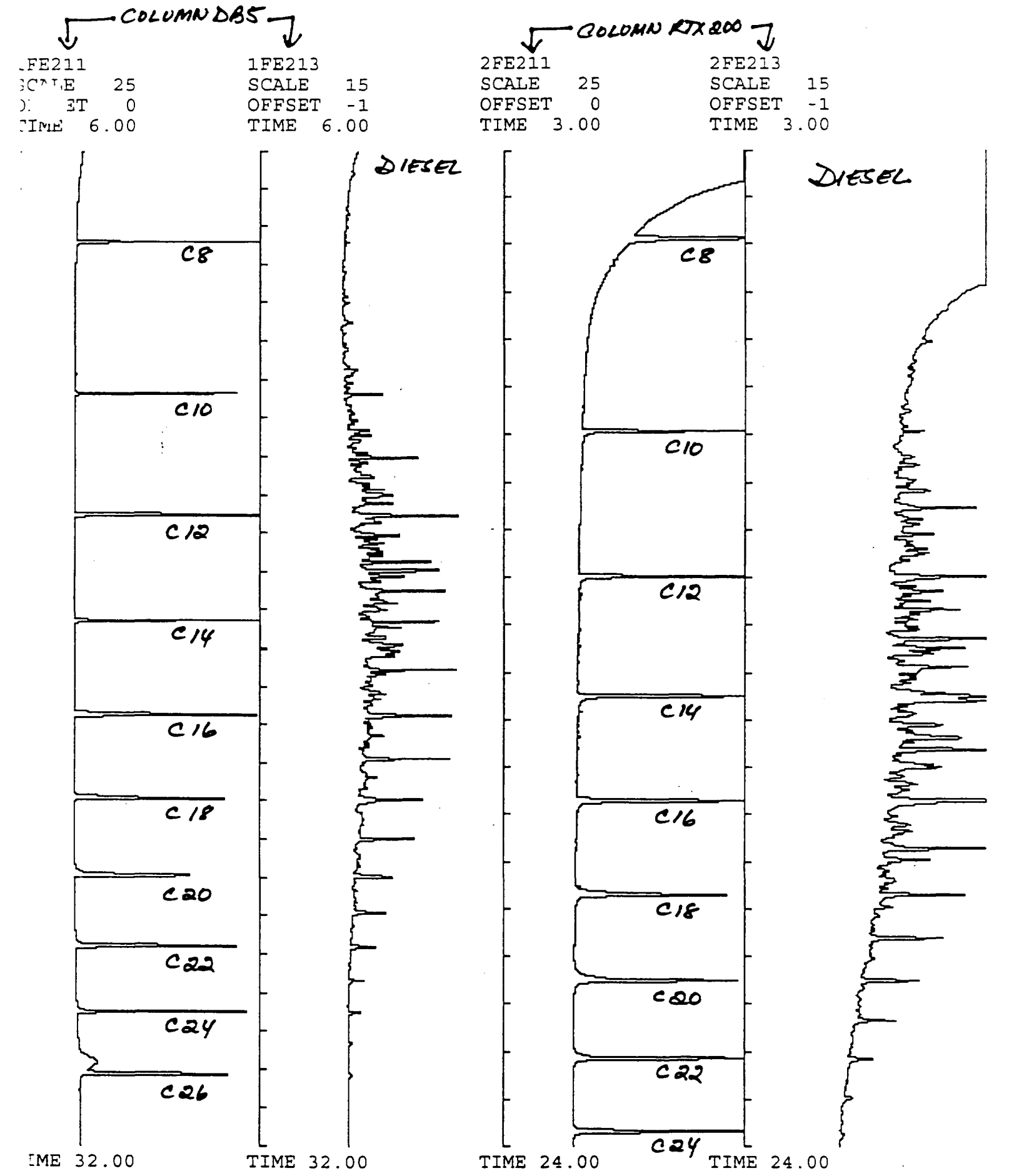
TABLE 1

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

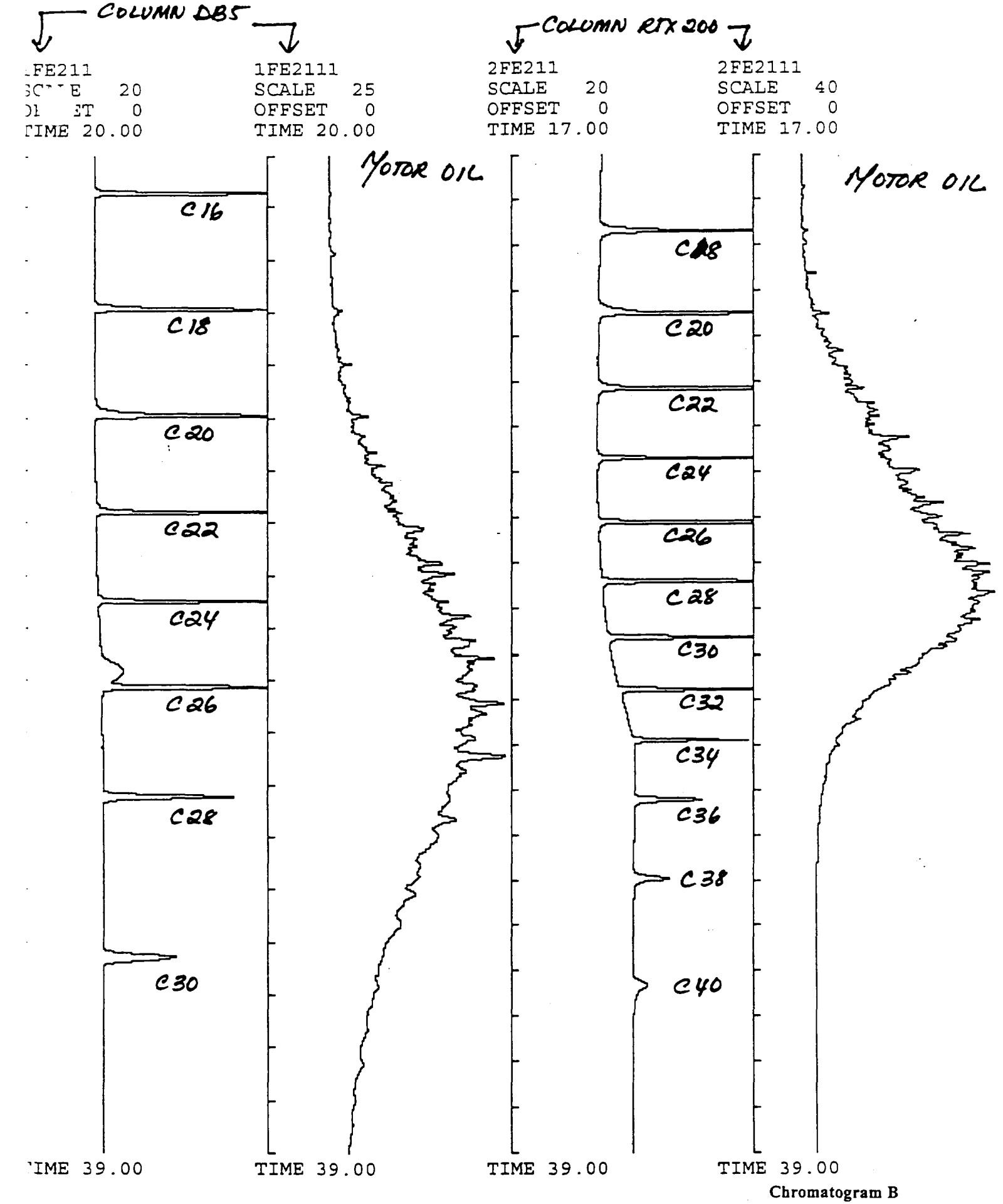
TABLE 2

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

SG70 APPENDIX – EXAMPLE CHROMATOGRAMS

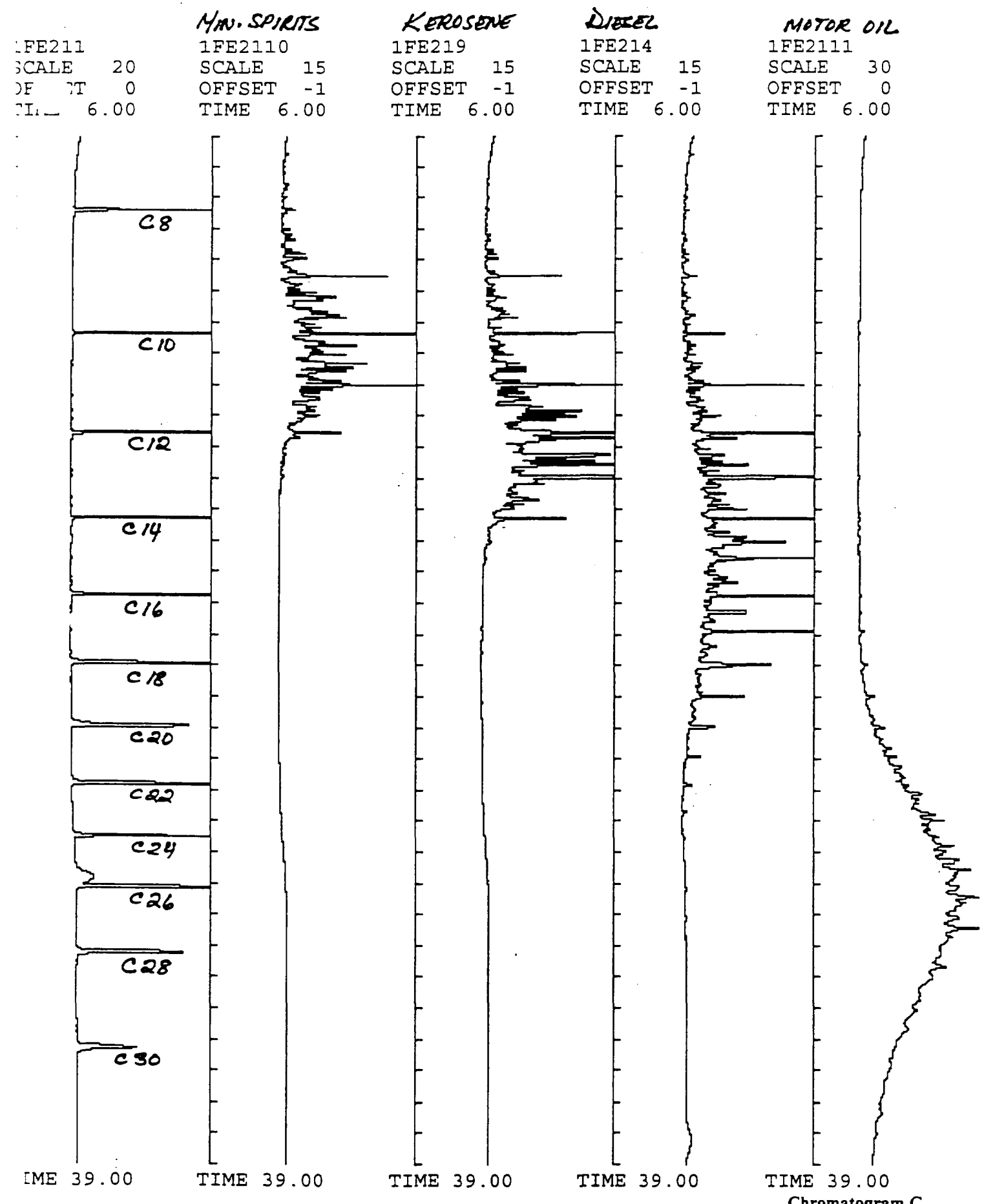


Chromatogram A

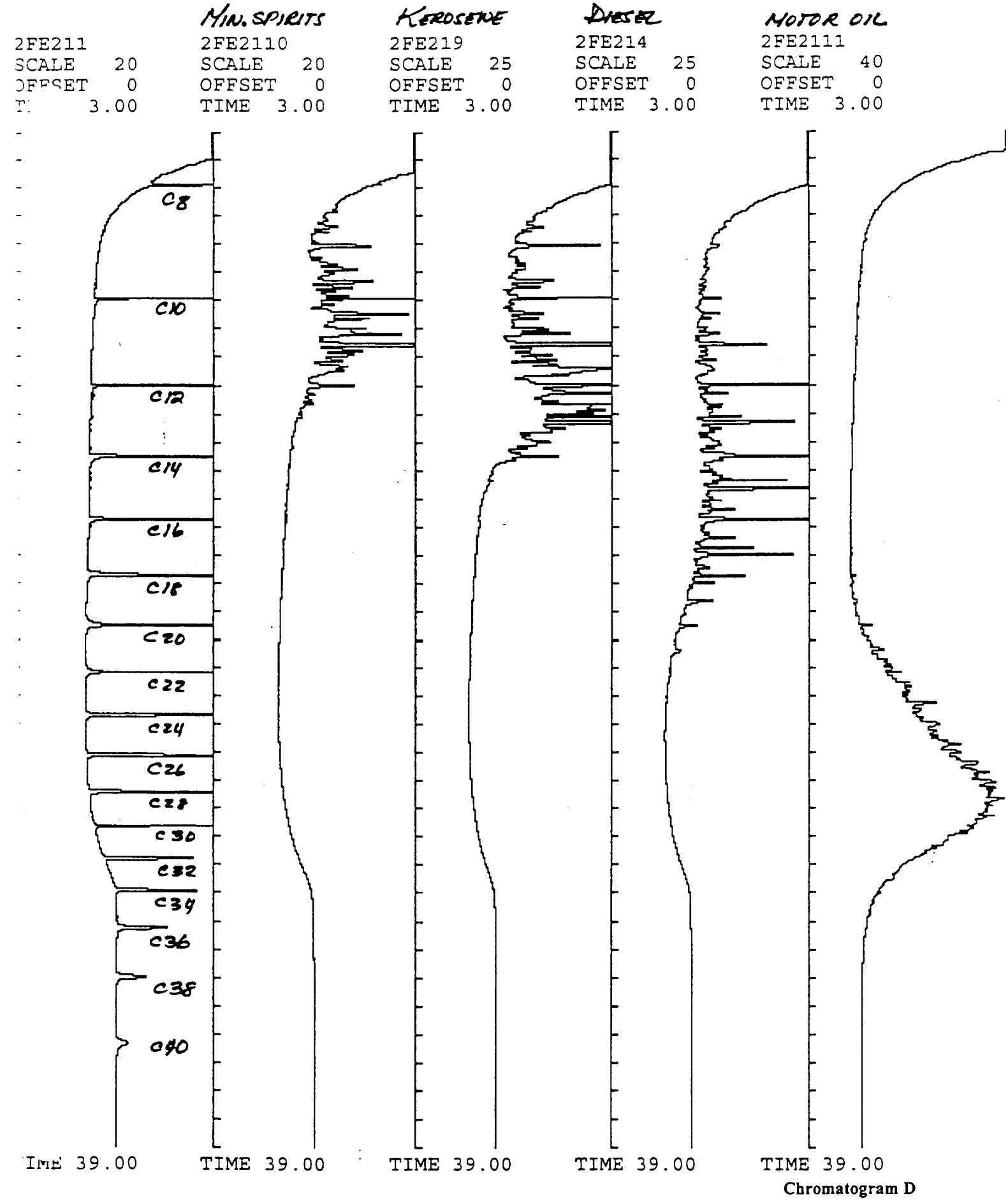


Chromatogram B

COLUMN DB5

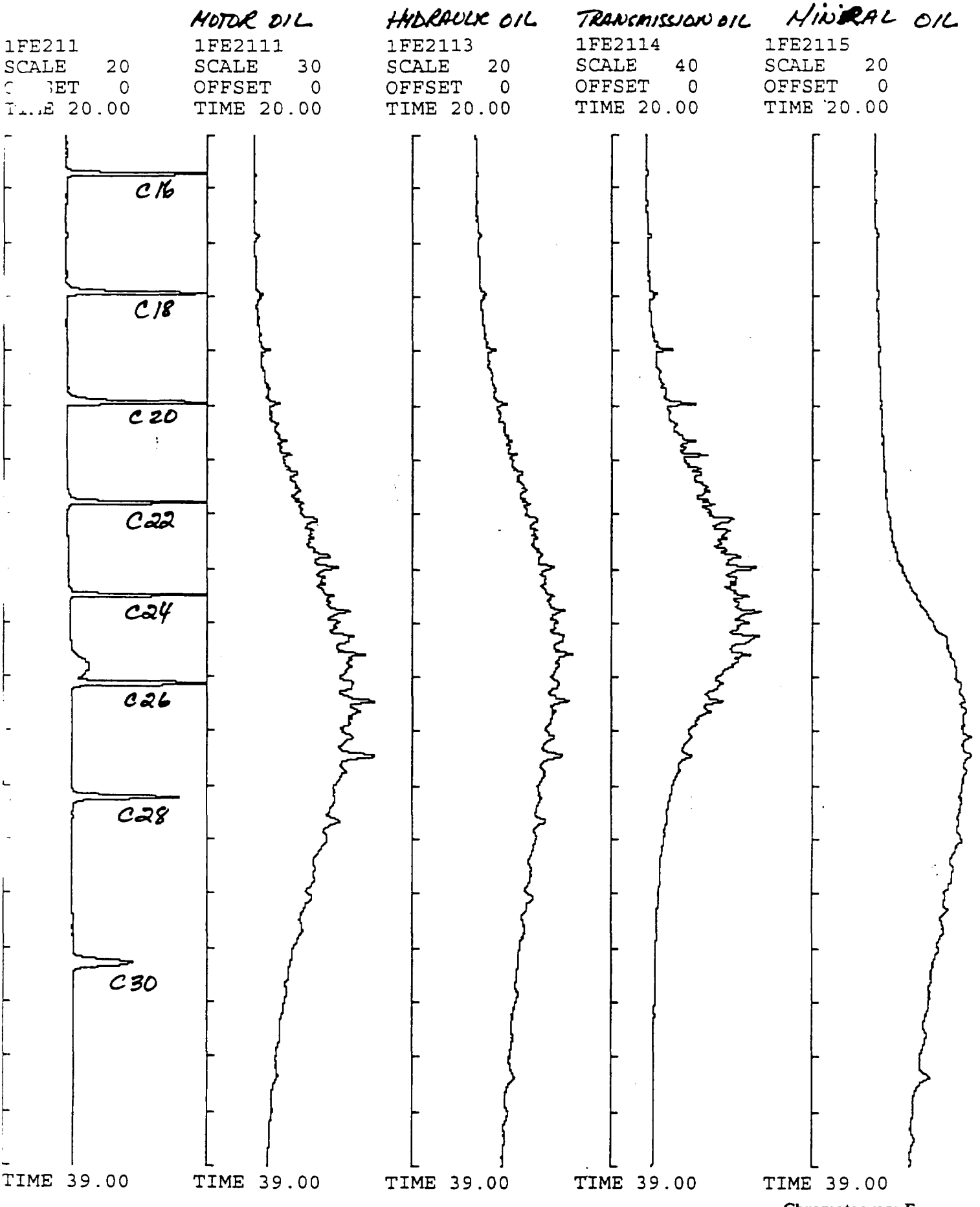


Chromatogram C



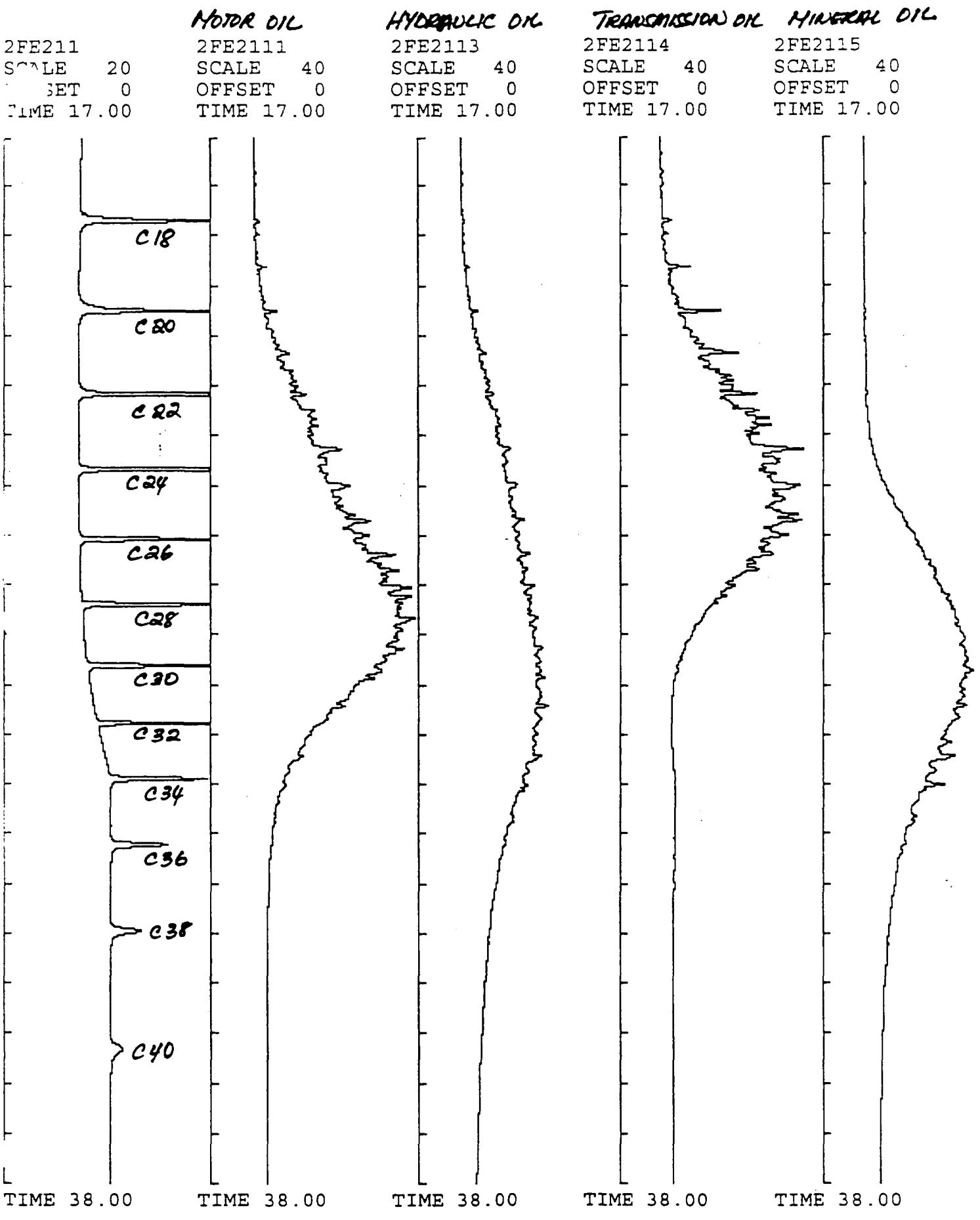
Chromatogram D

COLUMN DB5

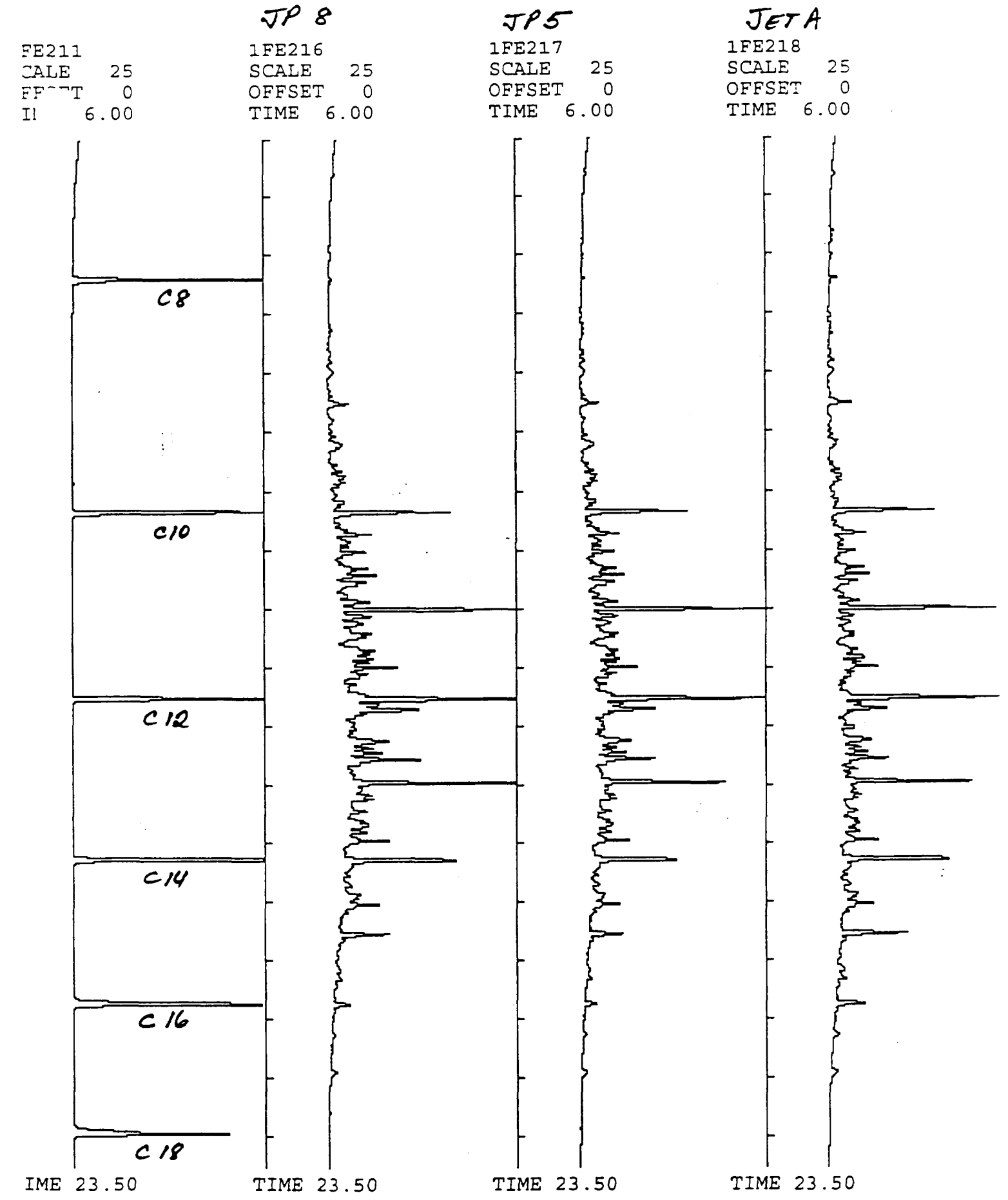


Chromatogram E

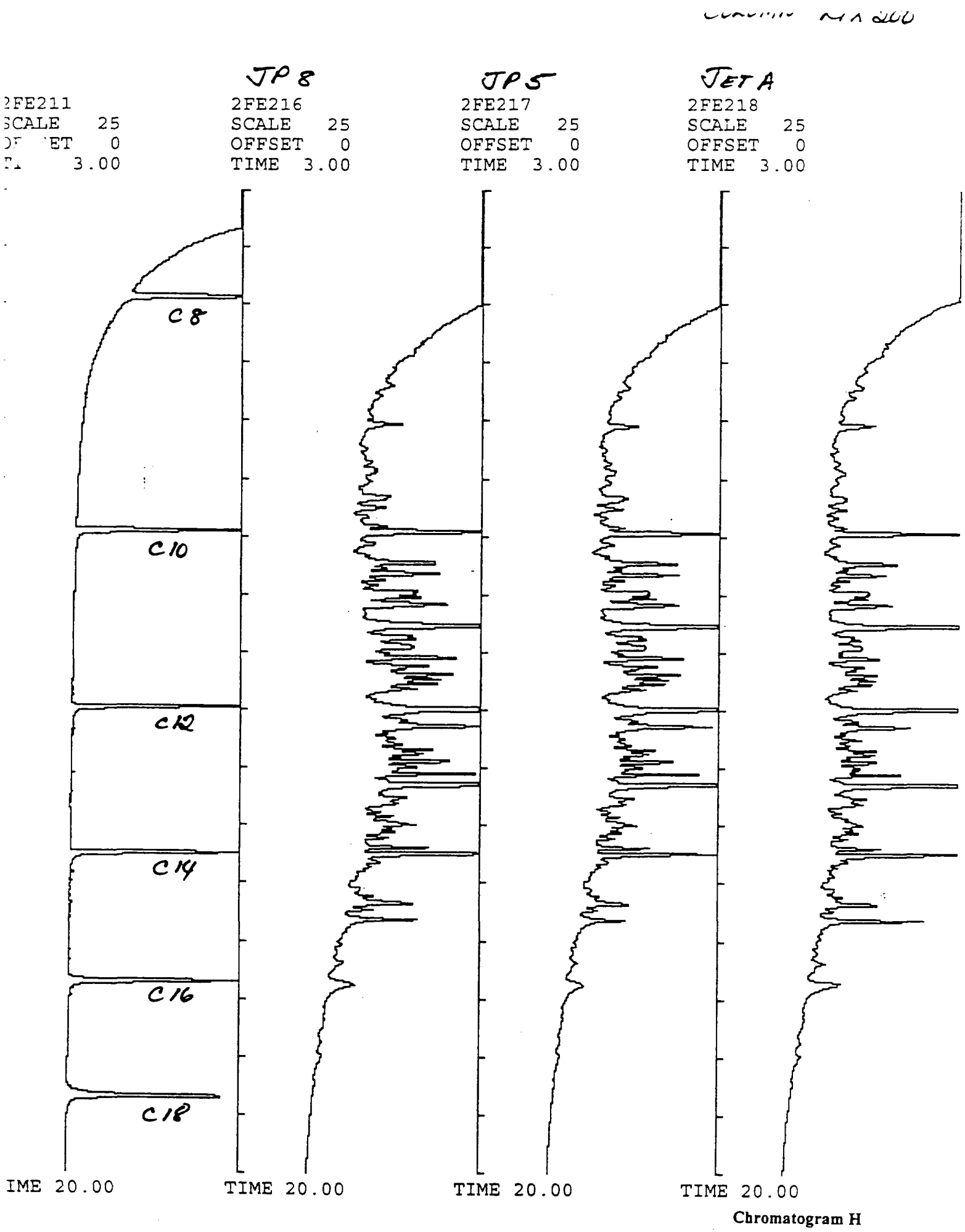
COLUMN RTX 200



Chromatogram F



Chromatogram G



Chromatogram H

COLUMN DB5

DIESEL

DIESEL #2 50% WEATHERED

1FE211
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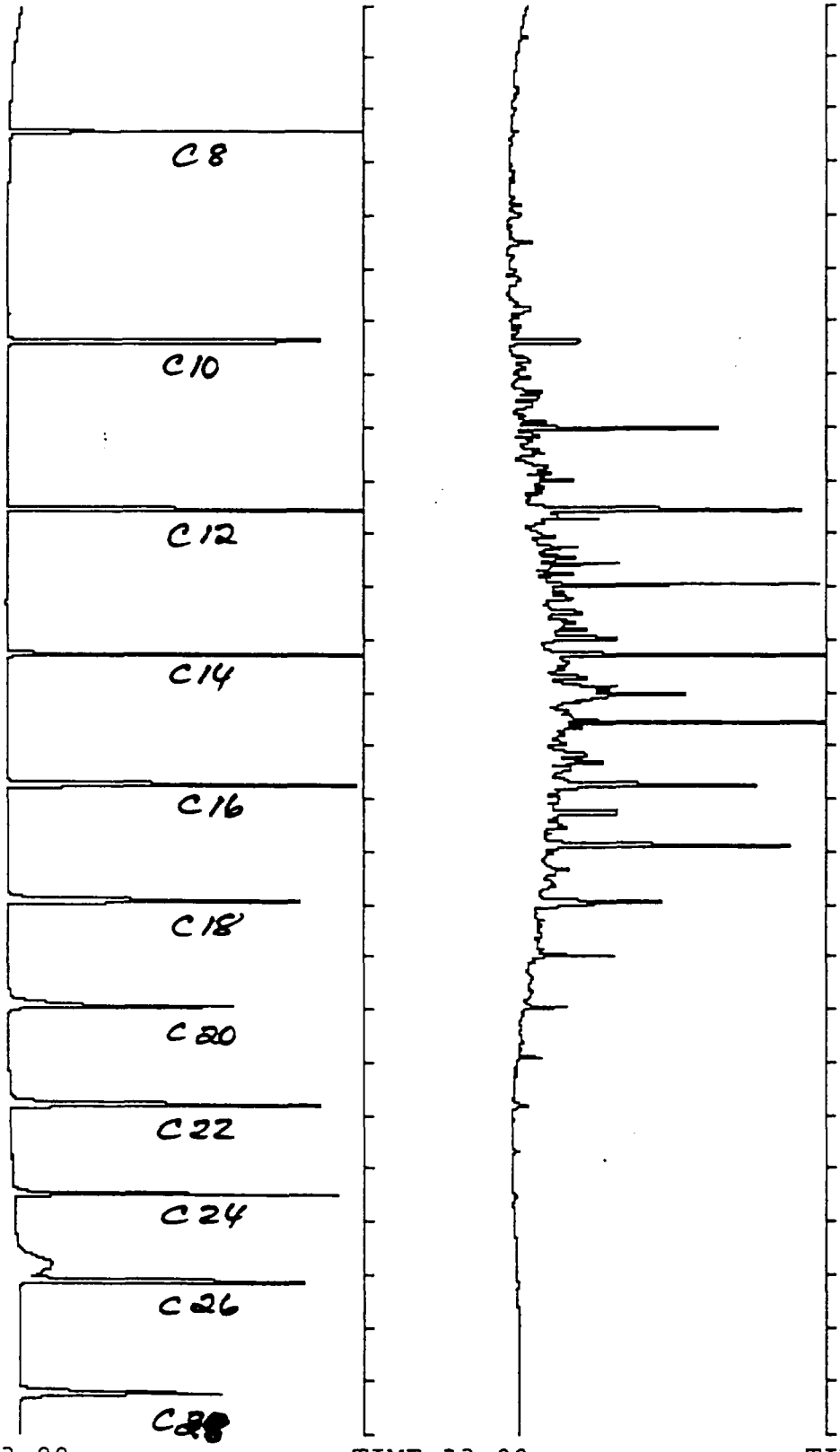
OFFSET 0
T. 6.00

1FE214
SCALE 20
OFFSET 0
TIME 6.00

1FE215
SCALE 25
OFFSET 0
TIME 6.00



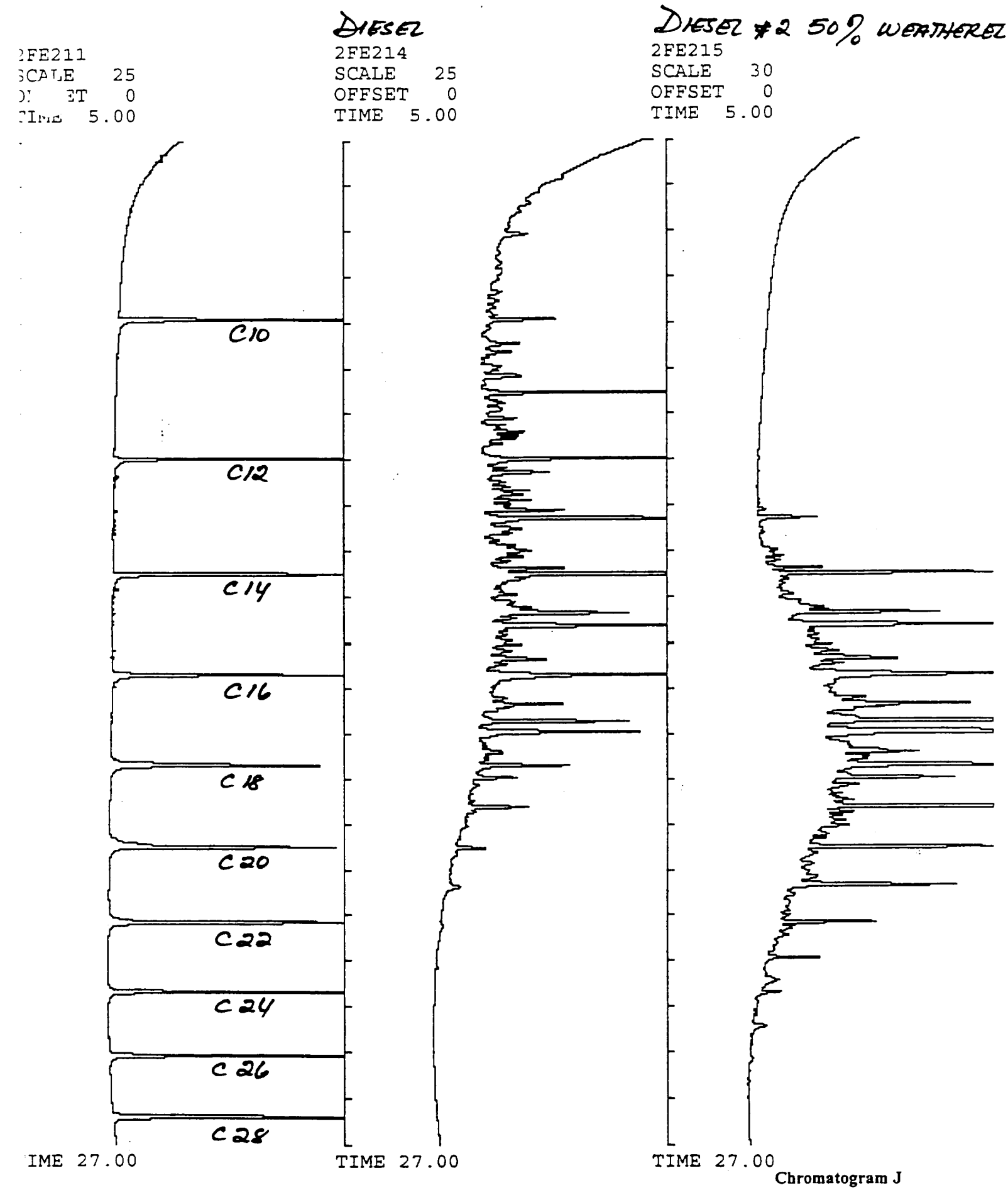
IME 33.00

TIME 33.00

TIME 33.00

Chromatogram I

COLUMN RTX200



Chromatogram J

COLUMN = DB5

TURPENTINE

NAPTHA

1FE211
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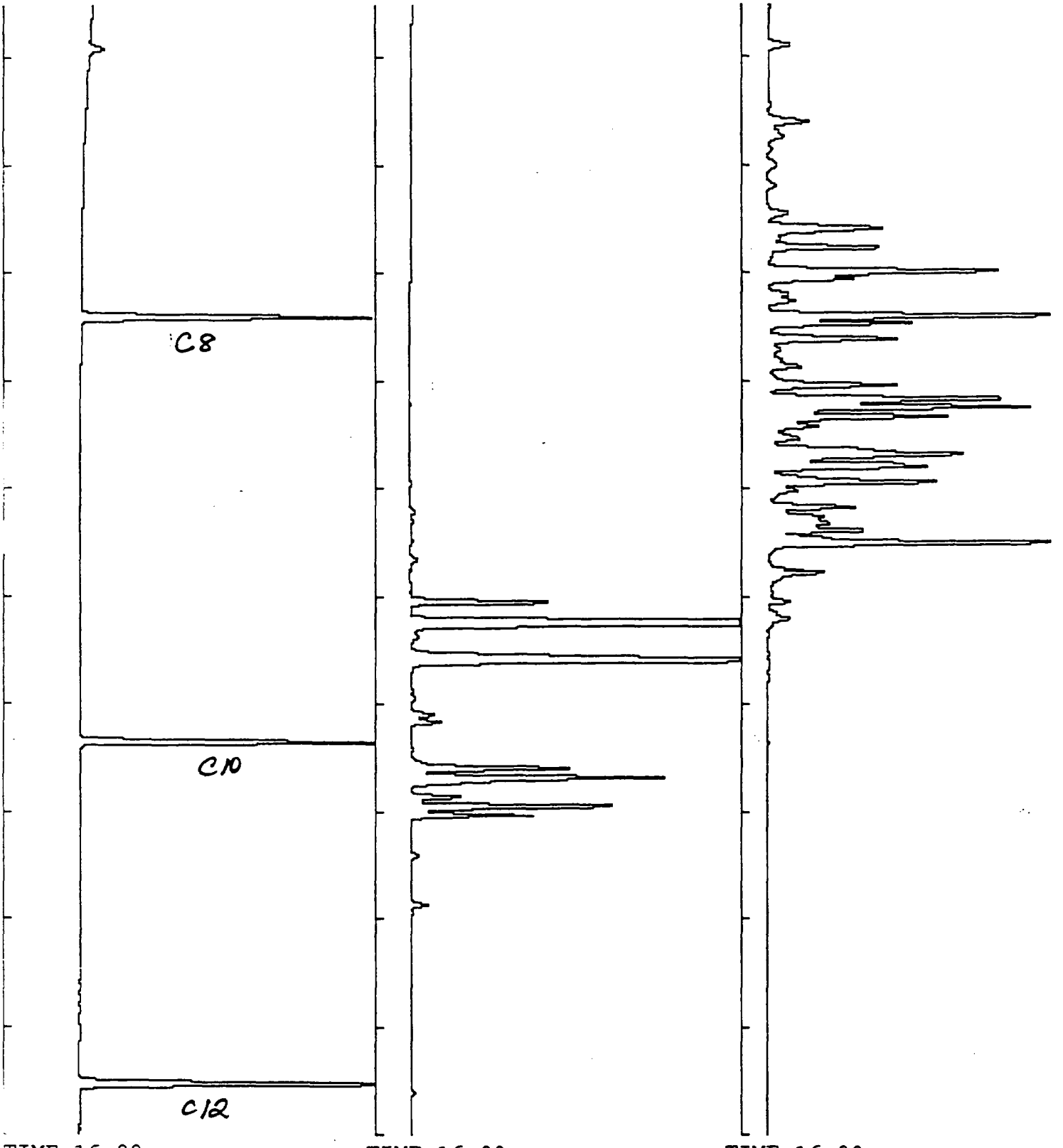
SET 0
TIME 5.50

1FE2117
SCALE 80
OFFSET 0
TIME 5.50

1FE2118
SCALE 125
OFFSET 0
TIME 5.50



TIME 16.00

TIME 16.00

TIME 16.00

Chromatogram K

COLUMN = RTX20.

TURPENTINE

NAPHTHA

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TIME 3.50

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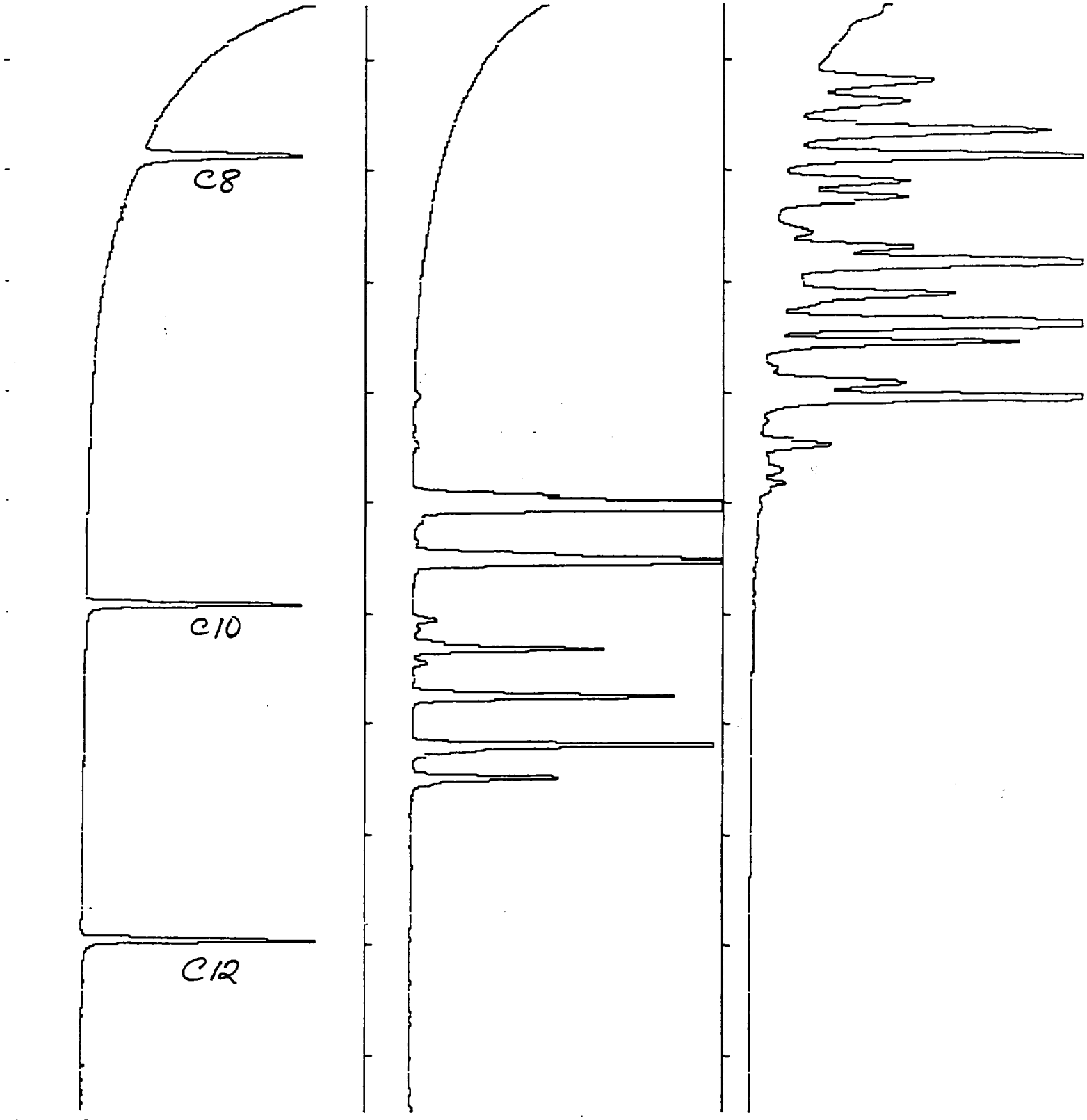
OFFSET 0
TIME 3.50

2FE2118
SCALE 150
OFFSET 0
TIME 3.50



IME 13.50

TIME 13.50

TIME 13.50

Chromatogram L

Approval Signature:	R. Wayne Robbins
Title: Corporate QA Manager	Date: 11/25/97

TOTAL CYANIDE AND CYANIDE AMENABLE TO CHLORINATION BY MANUAL DISTILLATION AND MANUAL OR AUTOMATED COLORIMETRIC DETERMINATION METHODS 335.1, 335.2, 335.3, 9010A, 9012, SM-4500-CN⁻C, SM-4500-CN⁻G

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of cyanide in drinking, surface and saline waters, aqueous wastes, leachates, sludges, oily waste, solids and soils. It is used to measure the concentration of both total cyanide and cyanide amenable to chlorination.

1.2 The Reporting Limit (RL) for both total cyanide and cyanide amenable to chlorination using this method is 0.010 mg/L for liquid samples and 1.0 mg/kg for soils using the manual colorimetric method. The reporting limit, method detection limit, accuracy and precision are listed in Table 5 of the Savannah Laboratories Comprehensive Quality Assurance Plan (1).

2.0 SUMMARY OF METHOD

2.1 A measured amount of sample is refluxed in a specially designed distillation apparatus. Cyanide, as hydrocyanic acid (HCN), is released as a gas by means of a reflux distillation operation under acidic conditions and trapped in a sodium hydroxide absorber solution. The cyanide in the absorbing solution is measured colorimetrically. The cyanide is converted to cyanogen chloride (CNCl) by reactions with chloramine-T at a pH<8 without hydrolyzing to the cyanate ion. After the reaction is complete, a red-blue colored complex is formed upon the addition of pyridine-barbituric acid reagent, and the absorbance is read at 578nm. The concentration of sodium hydroxide must be the same in the standards, the scrubber solutions, and any dilutions of the original scrubber solution, to obtain colors of comparable intensity.

2.2 This SOP includes a provision for automated cyanide color development using the Lachat analyzer included in Appendix 03. The automated procedure is capable of producing lower MDLs and Reporting Limits (RL) than the manual procedure if needed for client specific permit requirements.

2.3 When cyanide amenable to chlorination is to be determined, the sample is treated with calcium hypochlorite under alkaline conditions for one hour. After one hour, the excess hypochlorite is destroyed and the sample is analyzed for cyanide. The cyanide amenable to chlorination is the difference between the total cyanide and the cyanide remaining in the sample after treatment with hypochlorite.

2.4 Reduced sample volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method. This SOP is designed for 250 ml of aqueous sample, or aqueous sample diluted to 250 ml, or 3-5 g solid aliquot diluted to 250 ml so the distillation step can be semi-automated using a specially designed distillation apparatus.

2.5 This SOP is based on EPA Methods 335.1, 335.2, 335.3(2), 9010A, and 9012(3), and Standard Methods 4500-CN⁻C, 4500-CN⁻G(4). This SOP is in full compliance with EPA 335.2 which is approved for NPDES samples.

3.0 SAFETY

3.1 Cyanide is extremely poisonous. Avoid contact, inhalation or ingestion. Although very high levels of cyanide rarely occur in environmental samples, the analyst must be careful when handling these potentially hazardous and toxic samples.

3.2 The initial reaction product of alkaline chlorination is the toxic gas cyanogen chloride; therefore, the preparation of samples for cyanide amenable to chlorination must be done under a well ventilated hood.

3.3 The standards and reagents used in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standards are located in each laboratory. These sheets denote the type of hazards encountered, the safe handling instructions for these compounds, and first aid instructions.

3.5 Care must be taken when handling the distillation apparatus. Before handling glassware that has been used, allow the glassware to cool sufficiently for safe handling. The distillation flasks contain acids which can cause skin burns and destroy unprotected clothing.

3.6 The absorber solution contains sodium hydroxide which can cause skin burns and destroy unprotected clothing. This solution may also contain cyanide.

4.0 INTERFERENCES

4.1 Most interferences are eliminated or reduced using the incorporated distillation procedure.

4.2 Chlorine (residual) and other oxidizing agents when present, may cause a negative interference by decomposing cyanides during storage and manipulation. Chlorine should be destroyed at the time of sample collection using ascorbic acid; however, samples should be screened in the laboratory upon receipt to verify that no residual chlorine is present.

4.3 Sulfide adversely affects the colorimetric procedures for cyanide. The maximum holding time for NPDES permit compliance samples is 24 hours (collection to distillation) when sulfide is present (see 40 CFR Part 136.3 Table II Note 6)(5). For samples containing sulfides, the standard curve must be distilled in the same manner as samples. A sulfide scrubber containing lead acetate/acetic acid solution is placed in the distillation train to remove hydrogen sulfide just prior to the NaOH absorber as specified in EPA 335.2. Samples that contain hydrogen sulfide, metal sulfides or other compounds that may produce hydrogen sulfide under acid reflux should be distilled using the lead acetate/acetic acid scrubber. As a practical matter since this SOP is written for water, leachates, sludges and soil matrices which have some probability of containing sulfide, the lead acetate/acetic acid scrubber is incorporated and used on all samples along with the distillation of the standard curve, and all samples and standards are treated with an excess of bismuth nitrate to remove sulfide interference.

4.4 High cyanide results may be obtained for samples that contain nitrate and/or nitrite levels > 10 mg/L. During distillation nitrate and nitrite will form nitrous acid which will react with some organic compounds to form oximes which can decompose under test conditions to generate HCN. The interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid. Since this SOP incorporates the addition of bismuth nitrate to all samples, sufficient sulfamic acid is required to reduce this source of nitrate interference.

4.5 The presence of surfactant may cause the sample to foam during refluxing. If this occurs, the addition of an antifoaming agent will prevent the foam from collecting in the condenser. Fatty acids will distill and form soaps in the NaOH absorber solution. This and other sources of turbidity interference may be compensated using a color correction technique (see Appendix 02).

4.6 Some sulfur compounds (e.g. thiosulfate) may decompose during acidic reflux distillation, releasing sulfur, hydrogen sulfide (H_2S), and sulfur dioxide (SO_2). Sulfur compounds may convert cyanide to thiocyanate (SCN^-). H_2S and SO_2 may interfere with the colorimetric procedure for cyanide. Sulfur dioxide forms Na_2SO_3 which consumes chloramine-T reagent used to convert CN^- to CNCl in the colorimetric determination.

4.7 Other compatible procedures for removal or suppression of interferences may be employed provided they do not adversely effect the overall method performance.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

5.1 Soil samples are collected in wide-mouth plastic or glass jars. Soil samples are stored at 4 C ± 2 C until time of analysis and may be held up to 14 days from sample collection to distillation.

5.2 Water samples are collected in 500 ml or 1000 ml plastic or glass containers and are preserved by adding NaOH until sample pH $\geq$ 12 followed by cooling to 4°C ± 2°C. Samples known to contain residual chlorine are checked and chlorine neutralized in the field using ascorbic acid during sampling. A dark blue dot (color coded) on the sample bottle indicates the bottle was provided by SL with NaOH preadded to the bottle. Samples preserved with NaOH are maintained under refrigeration at 4°C ± 2°C for up to 14 days from sample collection to distillation. Upon arrival in the laboratory, the pH, sulfide and residual chlorine are checked on water samples to insure samples have been properly collected and preserved.

5.2.1 Pour a small aliquot into a plastic cup and test for chlorine (residual) using KI-starch paper. If the water sample contains chlorine, the paper turns a blue color. Add ascorbic acid a few crystals at a time and mix to reduce the chlorine. Continue adding ascorbic acid until the sample produces no color on the paper KI-starch indicator. Finally, add 0.6 g additional ascorbic acid for each liter of water. Record any adjustments and comments.

5.2.2 Pour a small aliquot into a second plastic cup and test for sulfide using lead acetate paper moistened with acetic acid solution and record results. The paper turns black/brown in the presence of sulfide. If the water sample contains sulfide and is an NPDES permit compliance sample, the distillation must be performed within 24 hours of sample collection. This is a regulatory holding time requirement for NPDES compliance samples. These samples will be assigned method 335.2 and project manager will verify NPDES compliance status. Any non-NPDES compliance sample with sulfide present should be distilled within 24 hours if possible or as soon as practical.

5.2.3 Check for pH using pH paper and record results. Add 10N NaOH solution to adjust pH $\geq$ 12 if required. Record any adjustments and comments.

6.0 APPARATUS AND MATERIALS

6.1 Reflux distillation apparatus consisting of a 500 ml flat bottom flask connected to a West-type condenser, scrubber, absorber, teflon tubing, connectors, neoprene rubber stoppers, air inlet, vacuum source, and hot plates. (See Figure 6.1)

6.2 UV-visible spectrophotometer set at 578 nm with 1 cm or larger glass cuvette. A flow through cell may be used for manual color development.

6.3 Volumetric flasks.

6.4 Volumetric pipets

6.5 25-ml buret

6.6 250-ml plastic cups

6.7 25-ml disposable pipets

6.8 Glass centrifuge tubes

6.9 Boiling chips

6.10 Spatulas

7.0 REAGENTS

7.1 Reagent water, lab generated deionized water

7.2 Calcium hypochlorite (Fisher cat #C100-500)

7.3 Potassium Iodide (KI)-starch paper (VWR cat #60799-008)

7.4 Sodium Arsenite - reagent grade (VWR cat #JT3487-4)

7.5 Lead Acetate paper (CMS cat #500-595)

7.6 Sulfamic Acid - reagent (1N) - (VWR cat #1931-500 x NY) Dissolve 50 g of sulfamic acid in deionized water and mix. Dilute to 500 ml. Prepare this reagent monthly.

7.7 Sodium hydroxide - reagent - (VWR cat #EM-SX0593-3)

7.8 Diluent solution (0.25N NaOH) Dissolve 10 g of sodium hydroxide in 800 ml of deionized water. Dilute to 1 liter. Prepare this reagent as needed. Used during color development to dilute absorber solution when using less than 25 ml solution (Section 10.2).

7.9 Sodium Hydroxide (10N) Measure 500 ml of deionized water into a 2 liter beaker. Place the beaker on a magnetic stir plate and add a teflon stir bar to the beaker. Weigh out 400 g of NaOH into a 1 liter beaker. Add a small quantity of the sodium hydroxide from the beaker to the deionized water. As the NaOH dissolves add more of the NaOH from the beaker until all 400 g has been added. Prepare this reagent as needed. SEE CAUTION BELOW.

7.10 Sodium Hydroxide Solution - Dissolve 1.6g NaOH in 1 liter of deionized water. This solution is used as a diluent for CN^- standardization only.

7.11 Sulfuric Acid (H_2SO_4) - reagent (VWR cat#2900 2.5 x NY)

7.12 Sulfuric Acid (H_2SO_4) Solution (18N) measure 500 ml of deionized water into a 2 liter beaker. Place the beaker on a magnetic stirrer with a teflon coated stir bar. Carefully and slowly add 500 ml of concentrated H_2SO_4 to the deionized water. Prepare this reagent as needed. SEE CAUTION BELOW.

!!!**CAUTION**!!!: Heat will be evolved as the sodium hydroxide or sulfuric acid mixes with water. These solutions will cause skin burns and destroy unprotected clothing.

7.13 Sodium Dihydrogen Phosphate (NaH_2PO_4) - reagent - (VWR cat # 7917-500 · NY) Dissolve 138g of sodium dihydrogen phosphate in 800 ml of deionized water. Dilute to 1 liter.

7.14 Magnesium Chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) - reagent (Fisher cat # 5958-2.5 x NY) Dissolve 510 g $MgCl_2 \cdot 6H_2O$ in 500 ml of deionized water and dilute to 1 liter.

7.15 Pyridine-Barbituric Acid reagent (Fisher cat #P368-500, Fisher cat #1933373) Prepare this reagent in the hood. Add 60 g barbituric acid to a 1 liter volumetric flask. Place the flask on a magnetic stir plate and add a teflon stir bar. Add enough deionized water to wet the acid. Turn on the stir plate and add 300 ml pyridine, mixing constantly. Slowly add 60 concentrated HCl. Continue to stir on the magnetic stirrer plate and allow the solution to cool to room temperature. Bring to volume with reagent water. Store reagent in an amber bottle. Prepare this reagent every 6 months.

7.16 Chloramine-T reagent (Fisher cat #01779-250) Dissolve 1.0 g of chloramine-T in deionized water and dilute to 100 ml. This reagent is made daily.

7.17 Ascorbic acid - reagent - (VWR cat #4407-500 x NY)

7.18 Silver Nitrate ($AgNO_3$) Titrant (0.0141N) (Fisher cat #SS82-1) Dissolve 2.395g of $AgNO_3$ with deionized water in a 1 liter volumetric flask and dilute to volume.

7.19 Sodium Chloride (NaCl) Solution (0.0141N) (VWR 7581-2.5 x NY) Dissolve 0.824 g of NaCl with deionized water in a 1 liter volumetric flask and dilute to volume.

7.20 Potassium Chromate indicator solution (Fisher cat #P220-100) Dissolve 50 g of K_2CrO_4 in deionized water. Add $AgNO_3$ solution until a definite red precipitate is formed. Let stand 12 hours, filter and dilute to 1 liter with distilled water.

7.21 I Indicator Solution - Dissolve 20 mg of p-dimethylamino-benzalrhodanine in 100 ml acetone.

7.22 Scrubber solution - Dissolve 30 g lead acetate trihydrate $Pb(C_2H_3O_2) \cdot 3H_2O$ + 6 ml acetic acid in 90 ml of distilled water. Adjust pH to 4.5 with acetic acid and dilute to 1 liter.

7.23 Absorber solution (0.5 N) - Dissolve 20g NaOH and 0.80g Sodium Arsenite to 1 liter deionized water. Prepare as needed.

7.24 Cadmium nitrate (VWR cat # 4008-125 x NY)

7.25 Bismuth nitrate ($Bi(NO_3)_3$) · 5 hydrate) Solution (0.062M) (Fisher cat #EK1938372) Dissolve 30 g $Bi(NO_3)_3 \cdot 5H_2O$ in 100 ml deionized water. While stirring, add 250 ml of glacial acetic acid. Stir until dissolved and dilute to 1 liter with deionized water.

7.26 Cyanide dilution water - Dilute 6 ml of 10 N NaOH and 1.8g ascorbic acid to 3.56 L with deionized water. This is used to dilute samples prior to distillation.

NOTE: All chemical reagent sources are subject to change depending on SL's yearly contract with various vendors.

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 in Inorganic Analyses.

8.1 Preparation of the Stock and Working Standards

8.1.1 Stock potassium cyanide (KCN) - reagent grade (Fisher cat #P223I-100) Cyanide Stock Standard (1000mg/L CN^-) - Dissolve 2.503 g of KCN and 2 g of NaOH in 800 ml of deionized water in a 1 liter volumetric flask and dilute to volume. Standardize with 0.0141 N $AgNO_3$ (1 mL = 1 mg CN^-) to determine actual CN^- concentration monthly.

8.1.2 Potassium cyanide Intermediate Standard (100 mg/L CN^-) Add 10 ml of the potassium cyanide stock solution (1000mg/L CN^-) to a 100 ml volumetric flask and dilute to volume with deionized water (1 mL = 100 μg CN^-). This standard is used to prepare the undistilled check standard/CCVS, MS/MSD and the calibration standards. Prepare on the day of standardization.

8.1.3 Stock sodium cyanide (NaCN) - reagent grade (Fisher cat #S284I-100) Cyanide Stock Standard (1000mg/L CN^-) - Dissolve 2.50g NaCN and 2 g NaOH in 800 ml of deionized water in a 1 liter volumetric flask and dilute to volume (1 mL = 1 mg CN^-). Standardize with 0.0141 N $AgNO_3$ (1 mL = 1 mg CN^-) to determine actual CN^- concentration monthly.

8.1.4 Sodium cyanide Intermediate Standard (100mg/L CN^-) Add 10 ml of the cyanide stock solution (1000mg/L CN^-) to a 100 ml volumetric flask and dilute to volume with deionized water (1 mL = 100 μg CN^-). This standard is used as an independent second source standard for LCS/LCSD. Prepare on the day of standardization.

NOTE: All standard sources are subject to change depending of SL's yearly contract with various vendors.

8.2 Standardization of $AgNO_3$ titrant is performed monthly.

8.2.1 Measure 100 ml of deionized water which is used as a blank.

8.2.2 Add 1 ml of potassium chromate indicator solution and titrate to a pinkish yellow end point. A blank of 0.2 to 0.3 ml is usual. Run in duplicate to verify and average results.

8.2.3 Dilute 10 ml of 0.0141N NaCl solution to 100 ml with deionized water.

8.2.4 Add 1 ml of potassium chromate indicator solution and titrate with $AgNO_3$ solution to pinkish yellow end point. Record the volume of $AgNO_3$ solution. Run in duplicate to verify normality and average results.

8.2.5 Calculation: The normality of $AgNO_3$ solution is calculated based on the following formula:

N of $AgNO_3$ = (N of NaCl x ml NaCl) ÷ Blank corrected ml of $AgNO_3$

8.3 Standardization of Stock Cyanide Standards (KCN & NaCN) performed monthly

8.3.1 Measure 100 ml of the NaOH solution (7.10)

8.3.2 Add 0.5 ml of the indicator solution (7.21) and titrate to the first change in color from a canary yellow to a salmon color is seen. Run in duplicate to verify and average results.

8.3.3 Titrate the primary stock standard (KCN) as in 8.14 using 10 ml of the 1000 mg/L KCN standard + 90 ml NaOH (7.10) solution.

8.3.4 Titrate the secondary stock standard (NaCN) as in 8.14 using 10 ml of the 1000 mg/L NaCN standard + 90 ml NaOH (7.10).

8.3.5 Calculations

each $Ag(CN)_2$ requires 2KCN based on the following reaction $Ag^+ + 2CN^- - [Ag(CN)_2.]^-$.

N of KCN = (2 x blank corrected ml of $AgNO_3$ x N $AgNO_3$) ÷ ml KCN

CN^- concentration (mg/L) =({N KCN x 26.03g} x 1000) ÷ 1 Liter

NOTE: The same procedure and equations apply for NaCN standardization.

8.4 Generation of the calibration curve: Methods 9010A, 335.2, 335.3 9012 and SM-4500-CN requires the distillation of the calibration standard curve for samples containing H_2S. Samples which do not contain H_2S can be analyzed using an undistilled calibration curve; however, a low and high standard needs to be distilled daily along with the samples to verify the distillation process can achieve ± 10% of the true value. The manual distillation of the standard curve and color development is a very time consuming process. Since samples may arrive on any given day containing H_2S requiring both 24 hour sample distillation and distillation of the standard curve, the distilled calibration curve is prepared in advance and is good for 30 days or until CCVS fails to meet ± 10% acceptance criteria. Standard are distilled in the same manner as samples (section 9.2). The undistilled check standard is generated daily and must meet the ± 10% acceptance criteria outlined in section 10.10.

Depending on the true concentration of KCN and NaCN determined after the monthly standardization, the final concentrations of the calibration standards may change slightly. An example for generating a calibration curve is listed in 8.4.1.

8.4.1 The KCN intermediate standard (100 mg/L) is used to prepare the calibration standards. Add the volumes of the KCN intermediate standard given in the table below to individual distillation flasks. Dilute to 250 ml with the CN^- dilution solution (7.26).

Volume of the 100 mg/L KCN Standard (mL)	Final Volume (mL)	Concentration of the CN^- Calibration Standard (mg/L)
0.000	250	0.000
0.040	250	0.016
0.100	250	0.040
0.200	250	0.080
0.300	250	0.120
0.400	250	0.160
0.500	250	0.200

These standards are distilled and color developed in the same manner as the samples.

8.5 Verification of the Calibration Curve (performed with each color development batch)

8.5.1 Prepare the undistilled check standard/CCVS from the KCN intermediate standard (100 mg/L) by pipetting 0.15 ml of the 100 mg/L KCN standard into a 100 ml volumetric flask containing 25 ml of diluent solution (7.8) (dilute absorber solution). Proceed with color development as in steps 10.3 through 10.8.

8.5.2 Assess recovery of the undistilled check standard/CCVS to determine if the calibration curve may be used for calculation of sample results. The recovery of the undistilled check standard/CCVS must be ± 10% of the true value.

9.0 SAMPLE PREPARATION AND DISTILLATION

9.1 Chlorination Step for Cyanide Amenable to Chlorination

9.1.1 Transfer 250 ml or an aliquot diluted to 250 ml of an aqueous sample or 3-5g of a soil, oil or sludge sample diluted with 250 ml of CN^- dilution solution (7.26) to a 500 ml beaker.

9.1.2 Place the beaker onto a magnetic stir plate in a fume hood. Add a Teflon stir bar to the beaker and stir the sample. Cover the sample with a box . This step must be performed in the dark since $K_3[Fe(CN)_6]$ may decompose under UV light and hence will test positive for cyanide amenable to chlorination if exposed to fluorescent lighting or sunlight.

9.1.3 Two sample aliquots are required to determine cyanides amenable to chlorination, one that has been chlorinated to destroy all amenable cyanide present and the other unchlorinated. Add approximately 0.5 g of calcium hypochlorite to one sample aliquot while stirring for the cyanide amenable to chlorination test. The pH should be maintained between 11 and 12 using 1.25 N NaOH solution during alkaline chlorination. Check the sample for hypochlorite using KI-starch paper and continue to add hypochlorite and maintain the pH until an excess of chlorine is present. Check the sample for chlorine every 15 minutes until the 1 hour alkaline chlorination is complete. Sample should be stirred continuously during chlorination procedure.

9.1.4 After 1 hour, add 0.5 g portions of ascorbic acid to the chlorinated aliquot until KI-starch paper shows no residual chlorine (paper is white). Add an excess of ascorbic acid to insure the presence of reducing agent. Add ascorbic acid to the second aliquot to be used for total cyanide so both portions will have the same approximate ascorbic acid level.

9.1.5 The sample is now ready to be distilled and analyzed for cyanide content. Distill both the chlorinated and unchlorinated aliquots as in the method for total cyanide (section 9.2). Distill and analyze both aliquots in the same batch if possible using the entire sample treated by alkaline chlorination. Calculations for cyanide are based on the original volume or weight measured at the start of the process.

9.2 Sample and Standard Distillation

9.2.1 Assemble the distillation apparatus as shown in Figure 1. Place 25 ml of 0.5 N NaOH into each absorber tube.

9.2.2 Place 28 ml of the lead acetate/acetic acid mixture in each scrubber tube. Make sure that the glassware is properly assembled and there are no leaks in the system. The lead acetate/acetic acid scrubber is placed in line just prior to the NaOH absorber.

9.2.3 Add 250 ml or an aliquot diluted to 250 ml of an aqueous liquid sample or 3-5 g of a soil, oil or sludge sample diluted to 250 ml with CN^- dilution solution (7.26) to the 500 ml flat bottom distillation flask. Record all volumes and weights in the log book.

9.2.4 Turn on the cooling water to the condensers.

9.2.5 Insert an inlet tube into each flask and turn on the vacuum. Adjust the flow so the scrubbers and absorbers are bubbling vigorously and air bubbles enter the boiling flask.

9.2.6 Add 25ml of bismuth nitrate solution to the sample through the inlet tube. This is to eliminate potential sulfide interference. Although samples are checked for free sulfide using lead acetate paper, bismuth nitrate

is used to eliminate complexed sulfide released during acid reflux from the solids sample matrix.

9.2.7 Add 10 ml of 1 N sulfamic acid through the inlet tube, to eliminate nitrate interferences. Mix for three minutes. Excessive use of sulfamic acid could create a method bias.

9.2.8 Slowly add 25 ml of 1:1 H2SO4 to each sample through the inlet tube. Mix the sample while adding H_2SO_4 solution. Rinse the tube with deionized water and allow the air flow to mix the flask contents for 3 minutes.

9.2.9 Turn the hot plates on high.

9.2.10 Add 10 ml of the magnesium chloride solution to each flask through the inlet tube.

9.2.11 When condensate begins to drip back into the flask from the condenser begin timing for 1 hour.

9.2.12 After 1 hour, turn the hot plates off and let the system cool with the air flowing through the system for 20 minutes.

9.2.13 Turn off the vacuum and the water flow to the condensers. Remove the absorber tubes from the apparatus.

9.2.14 Rinse the absorber inlet tube several times with deionized water. The rinsate is added to the absorber tube and diluted to 50 ml final volume with deionized water and color developed as soon as convenient or no later than 72 hours after the distillation. Distillation of the samples must be initiated within 14 days of sample collection.

10.0 MANUAL COLOR DEVELOPMENT PROCEDURE

10.1 Turn on the spectrophotometer and allow it to warm up for 30 minutes before absorbance readings are taken. Set the wavelength to 578 nm.

10.2 Pipet 25 ml of each sample distillate, or sample diluted to 25 ml with diluent solution (7.8) into a 100 ml volumetric flask containing 25 ml of deionized water. This includes the samples, LCS/LCSD, MSD/MSD and the Method Blank. The undistilled check standard/CCVS is also prepared as in step 8.5.1, at this time.

10.3 Pipet 15 ml sodium phosphate buffer solution to each flask.

10.4 Pipet 2.5 ml of the Chloramine-T solution to each flask and mix well.

10.5 Let stand for one minute.

10.6 Test for the presence of chlorine with KI-starch paper. Add additional chloramine-T if necessary.

10.7 Add 5 ml of the pyridine-barbituric acid solution to each flask and dilute to 100 ml with deionized water. Allow 8 minutes for color development.

10.8 Zero the spectrophotometer with the cuvette or flow through cell filled with continuing calibration blank at the beginning of each run (and after every 10 samples). Then read the absorbance of the sample distillates on the spectrophotometer. All absorbance measurements must be made within 15 minutes of color development. The color will range, depending on the concentration of cyanide present, from yellow to violet.

10.9 Starting with the CCVS pour the test solution into a clean 1-cm cuvette and place into the sample holder of the spectrophotometer. Read and record the absorbance on the cyanide analysis log. Continue reading and recording the absorbances of the standards and samples. A flow through cell system can also be used on the spectrophotometer.

10.10 The samples and standards must be analyzed in this specific order. The analysis sequence is:

- undistilled continuing calibration blank (CCB)
- undistilled calibration verification standard (mid-level) (CCVS) must be recovered at ± 10% of true value

- 10 client samples or QC item readings
- undistilled continuing calibration blank (CCB)
- undistilled continuing calibration verification standard (CCVS) must be recovered at ± 10% of true value
- 10 client samples or QC item readings
- Complete the sequence with a CCB and CCVS

CCB's and CCVS's are not distilled but contain all of the reagents used for color development.

10.11 Evaluate the Calibration Curve

10.11.1 Using a calculator or a PC capable of linear regression, enter either the concentration values(mg/L) or ug of CN^- added (x-axis values) and the corresponding absorbance values (y-axis values). The correlation coefficient of the calibration curve must be greater than or equal to 0.995 to determine the slope of the curve. If this criterion is not met, consult the immediate supervisor for help in determining the cause of the failure. If the problem cannot be resolved, the calibration curve must be prepared, analyzed and evaluated again. This means that all sample and QC item distillates associated with the calibration standards will have to be reanalyzed.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 After the slope of the curve is determined calculate the results of the samples as follows in sections 11.2 or 11.3:

11.2 Using the amount of CN^- (ug) vs. absorbance:

11.2.1 Determine the ug of CN^- in each sample:

ug CN^- = absorbance of samples x slope of curve

11.2.2 Determine the mg/L of CN^- in each sample:

Liquids: mg/L CN^- = [(ug CN^- x FV) ÷ C] ÷ S

FV = Final volume absorber solution (50ml)
C = Volume used for color development (ml)
S = Sample size distilled (ml)

Soils: mg/Kg dw CN^- = {[(ug CN^- x FV) ÷ C] ÷ S} ÷ Solids
FV = Final volume absorber solution (50 ml)
C = Volume used for color development (ml)
S = Sample size distilled (g)
Solids = Decimal equivalent of the percent total solids ("Solids" = percent total solids ÷ 100%)

For example, if the percent total solids of a sample is 85%, the Solids = 0.85; if the total solids is 100%, the solids = 1.0; if the percent total solids is 8.4%, the solids = 0.084.

11.3 Using concentration vs. absorbance:

Liquids: mg/L CN^- = (Abs ÷ Slope) x DF

ABS = absorbance determined on spectrophotometer
Slope = slope determined from calibration curve
DF = Dilution factor (if any)

Soils: mg/Kg CN^- = [(Abs ÷ Slope) x (0.25 + IW)] ÷ Solids

Abs = absorbance determined on spectrophotometer
Slope = slope determined from calibration curve
IW = Initial weight (Kg)
Solids = Decimal equivalent of the percent total solids. ("Solids" = % Total Solids ÷ 100%)

For example, if the percent total solids of a sample is 85%, the solids = 0.85; if the percent total solids is 100%, the solids = 1.0; if the percent total solids is 8.4%, the solids = 0.084.

11.3.1 Soils are calculated on a dry weight basis unless specified by the client.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of 1 to 20 client samples and the associated QC items that are distilled and analyzed together. The QC items for an analytical batch consist of a method blank, a lab control standard (LCS), and lab control standard duplicate (LCSD), a matrix spike (MS),a matrix spike duplicate (MSD) which are distilled. A continuing calibration verification standard (CCVS) and ICB and continuing calibration blank (CCB) are also analyzed undistilled.

12.2 Evaluation of QC Data

12.2.1 The method blank is reagent water that is taken through all distillation and analytical procedures. The concentration of cyanide in the method blank is a measure of the contamination due to the entire distillation and analytical process.

12.2.1.1 The concentration of cyanide in the method blank must be less than 0.010 mg/L for liquids and 1.0 mg/kg for soils.

12.2.1.2 If the concentration of cyanide in the method blank is greater than 0.010 mg/L for liquids and 1.0 mg/kg for soils, contact the immediate supervisor to determine the cause of the contamination. If the cause of the contamination cannot be determined and corrected, the batch of samples processed with the method blank must be analyzed again.

12.2.2 The LCS/LCSD are standards obtained or prepared from a separate source (a source different from the source of the calibration standards). The standard is NaCN.

12.2.2.1 The recoveries (%REC) of the LCS/LCSD must be between 85%-115%. The relative percent difference (%RPD) between the LCS/LCSD must be less than 30%.

12.2.2.2 If the recovery of cyanide in the LCS or LCSD is outside of the 85-115% range, contact the immediate supervisor to determine the cause. If the cause of the failure of either the % REC or %RPD cannot be determined and corrected, the batch of samples processed with the LCS/LCSD must be distilled and analyzed again.

12.2.3 The matrix spike and matrix spike duplicate (MS/MSD) is prepared by adding a known concentration of the 100 mg/L KCN standard to a known volume of a sample. The spiked samples are processed through the entire distillation and analytical procedure. The accuracy and precision results from the MS/MSD are a measure of the effect that the sample matrix has on the determination of cyanide.

12.2.3.1 The recoveries (%REC) of the MS/MSD should be between 75-125%. The %RPD for the MS/MSD should be less than 30%.

12.2.3.2 If the recovery of cyanide in the MS/MSD is outside of the 75-125% range, contact the immediate supervisor to determine the cause. If the %RPD of the MS/MSD is greater the 30%, contact the immediate supervisor to determine the cause. If the cause of the failure of either the %REC or % RPD cannot be determined and corrected and the LCS/LCSD are within the specified control limits, the cause can be attributed to matrix interference(s).

12.2.4 The undistilled check standard/CCVS must be within 10% of the true value of the KCN standard at the beginning of the batch, after every 10 samples and the at the end of the batch.

12.2.5 The method detection limit (MDL) is defined as the concentration of an analyte that can be measured with a 99% confidence that the result is greater than zero. The MDL is determined by spiking 7 to 10 aliquots of reagent water or 7 to 10, 3-5 g aliquots of blank soil (a soil that has been analyzed previously and found to contain no CN^-) with a known concentration of CN^-. The spiked samples are taken through the entire distillation and analytical process. The results of the analysis are used to calculate the MDL. A procedure is given to eliminate outliers from the MDL calculation. MDL studies are performed annually. When a new chemist/analyst begins work on cyanide an initial demonstration of laboratory capability (IDLC) must be performed consisting of a minimum of 4 replicates based on LCS/LCSD spiking levels with accuracy and precision achieved within SL QA Plan criteria.

12.2.6 Prepare the spiked samples for the determination of the MDL study.

12.2.6.1 Spike 7 to 10,250 ml aliquots of reagent water with 0.025 ml of the cyanide matrix spiking solution (100 mg/L; Section 8.1.2). The concentration of cyanide in the water sample is 0.010 mg/L for the manual color development procedure.

NOTE: For projects which require Florida DEP QAS criteria, matrix spike results will be utilized for laboratory control. If the matrix spikes are out of control, laboratory control standard and method control criteria will be utilized for ultimate control of the analytical batch.

13.0 PREVENTIVE MAINTENANCE

No guidance available in this revision.

14.0 TROUBLE-SHOOTING

No guidance available in this revision.

15.0 REFERENCES

15.1 Savannah Laboratories Comprehensive Quality Assurance Plan, and Corporate Quality Assurance Plan, current revisions.

15.2 Methods for Chemical Analysis of Water and Wastes: U.S. EPA Office of Research and Development: Cincinnati, OH, March 1983 EPA 600/4-79-020 Revised march 1983. EPA method 335.1, 335.2, 335.3.

15.3 Test Methods for Evaluating Solid Waste, SW-846, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., Update I, II and III, September 1994. EPA method 9010A, 9012.

15.4 Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition, APHA; Washington, D.C., 1992. SM methods 4500-CN^-C, SM-4500-CN^-G, SM-4500-CN^-C, SM-4500-CN^-G

15.5 Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office, Washington D.C., July 1, 1993.

APPENDIX 01.1
Field Method for Checking and Removing Residual Chlorine

Oxidizing agents, such as chlorine decompose most cyanides during storage and therefore should be removed at sample collection if known to be present. Collect water sample in 500 or 1,000 ml bottle. Check for the presence of chlorine using KI-Starch paper. If the water contains chlorine, the paper turns a blue color. Add ascorbic acid until the sample produces no color on the KI-Starch paper indicator. Finally, add 0.6 g additional ascorbic acid for each liter of water sample. Water samples which produce a negative chlorine test do not require addition of ascorbic acid. Add 2 NaOH pellets after chlorine is destroyed and check pH using pH paper or pH meter. Additional NaOH may be required to adjust pH $\geq$ 12. Record any adjustments or comments in field notebook. Samples which are not chlorinated (e.g. groundwater) do not require field test for chlorine.

Drinking water samples can be collected for cyanide using bottles prepared in the laboratory with ascorbic acid and NaOH pellets preadded to the sample bottles.

APPENDIX 01.2
Field Method for Detecting Hydrogen Sulfide (H_2S)

Sulfide may adversely affect the colorimetric procedures for cyanide and can be removed in the field or in the laboratory during distillation. Collect water sample in 500 or 1,000 ml bottle. Check for the presence of sulfide using moistened lead acetate paper. The paper darkens, turns black/brown, in the presence of sulfide. If the water sample contains sulfide and is a NPDES permit compliance sample, the distillation must be performed within 24 hours of sample collection. This is the regulatory holding time requirements for NPDES compliance samples. Notify the project manager if a sample contains sulfide to coordinate the holding time requirement or how to manager potential interference from sulfide.

APPENDIX 01.3
Field Method for Removing Hydrogen Sulfide (H_2S)

Water samples thought to contain sulfide should be tested for the presence of sulfide in the field using lead acetate paper before addition of NaOH for pH adjustment. If sulfide is present, it can be removed by complexing with cadmium nitrate powder until a negative sulfide test is obtained, followed by filtration to remove CdS precipitate. Sodium hydroxide (NaOH) is then added to the filtrate to adjust the pH $\geq$ 12. These steps must be performed on water samples in the field with sulfide precipitation, filtration and NaOH addition to pH $\geq$ 12 in this order. Since cadmium nitrate produces a potentially hazardous waste, it's use in removing sulfide by SL personnel is recommended only on NPDES samples containing sulfide in situations where the samples cannot be distilled within 24 hours of collection. The filtration step removes insoluble complexed cyanide compounds; therefore, if water samples contain particulate matter this solid fraction must be removed by filtration and analyzed separately as insoluble CN^- or removed by filtration and added back to the filtrate before distillation to measure total cyanide. Perform field sulfide removal only upon instructions of the project manager.

APPENDIX 01.4
Field Method for pH Adjustment of Samples Collected for Cyanide

Cyanides are very reactive and unstable; therefore, adjust pH ≥ 12 and cool at time of collection. If samples are known to be free of chlorine and sulfide, sample bottles can be prepared by adding NaOH pellets to empty bottles. Check pH to insure pH ≥ 12 using pH meter or pH paper. Add additional NaOH to adjust pH ≥ 12.

APPENDIX 02
Color Correction Procedure for Manual Color Development

Color and turbidity may interfere with the colorimetric determination of the sample. To correct for these interferences two sample aliquots will be used for color development. For aliquot (A) follow all steps in section 10 for color development. For aliquot (B) follow all steps in section 10 excluding step 10.4, the addition of chloramine-T. Calculate mg/L CN^- in both aliquots as in section 11.0.

$$mg/L\ CN^- = A - B$$

APPENDIX 03
Cyanide Color Development: Lachat QuickChem AE Procedure

SCOPE AND APPLICATION

This automated method for cyanide determination is applicable to absorber solutions distilled from ground water, drinking water, Surface water, domestic and industrial wastes, sediments, and soils. The practical quantitation limits for this method are 0.010 mg/L in liquids and 1.0 mg/kg in soils.

SUMMARY OF METHOD

This procedure utilizes reaction of cyanide with Chloramine-T to form cyanogen chloride which then reacts with the pyridine-barbituric reagent to give a red-blue colored complex. This SOP is based on EPA Method 335.3, 9012 and Lachat Method 10-204-00-1-A.

INTERFERENCES

Preliminary distillation with sulfide scrubbing will eliminate sulfide, turbidity and salt effects. Volatile organic acids, however, may carry over to the absorber. Their turbidity may be compensated for via color correction techniques.
Carbon dioxide, (CO_2) liberated from carbonate containing samples, causes bubbles during analysis on the Lachat auto analyzer. Calcium hydroxide is added to the absorber solution to precipitate the CO_2 as calcium carbonate, which is either settled or filtered to remove interference.

APPARATUS AND MATERIALS

QuickChem AE autoanalyzer:
- Automatic Sampler
- Proportioning pump
- Cyanide Reaction Module
- DQM Software

APPARATUS AND MATERIALS (Continued)

Disposable 50-mL polypropylene centrifuge tubes with flat caps
Filters, 0.45 um

Volumetric flasks: 100, 200, 250 mL

Pipettes: 20, 50, 100 uL

Autoanalyzer tubes, 13 mm diameter by 100 mm long

Scintillation vials (20-mL) with foamed polyethylene lined with polypropylene caps

REAGENTS AND STANDARDS

Note: Unless otherwise specified, all reagent chemicals and standards are Analytical Reagent grade (AR or ACS). When this criterion is met, specific supplier is not a consideration. A list of current suppliers is given in appendix B. These may change as Purchasing identifies competitive suppliers.

Water is deionized, Type II.

Calcium Hydroxide: Powder.

Carrier: (Diluted Absorber Solution)

Dissolve 10 g sodium hydroxide (NaOH) and 0.4 g sodium arsenite ($NaAsO_2$) in water and dilute to 1 L. Store up to 1 month in polypropylene.

Phosphate Buffer, 9.71 M:
Dissolve 97 g anhydrous potassium dihydrogen phosphate ($K_2H_2PO_4$) in water. Dilute to 1 L. Store in brown glass in the refrigerator up to 1 month.

Chloramine-T: Dissolve 2 g Chloramine-T hydrate in 500 mL water. Storage life is only 1 week.

Pyridine-Barbituric (Py-Bar) Acid Reagent: Either dilute manual method Py-Bar 1 + 3 with water or prepare as follows: Disperse 15 g barbituric acid in 100 mL water in a covered 1-L jar. When thoroughly dispersed, add 75 mL pyridine. Continue stirring for at least 2 hr, preferably over night. Add 15 mL concentrated hydrochloric acid (12 M HCl) and continue stirring for at least 1 hr. Transfer to a 1 L brown glass bottle and dilute to the base of the neck. Store in the refrigerator only 2 weeks. (Due to the limited stability of diluted Py-Bar, it is advisable to dilute only enough manual method Py-Bar to make the day's run on the analyzer. Typically 100 mL of manual method Py-Bar diluted to 400 mL is adequate.)

The primary stocks for standards are the ≈ 1000 mg/L stock CN^- for 335.2 and the ≈ 100 mg/L working stock. The calibration standards shown below are made by diluting the appropriate stock with carrier an are stable for 1 week.

TABLE I. CALIBRATION STANDARDS

Stock Concentration: 949 mg/L
Hundred Stock: 949.1 mg/L

Standard	Standard Used (mg/L)	Volume of Standard (mL)	Final Volume (mL)	Conc. CN^- (mg/L)
A	949	0.500	100	4.74500
B	94.9	0.500	25	1.89800
C	949	0.100	100	0.94900
D	94.9	0.100	25	0.37960
E	Standard A	1.000	25	0.18980
F	Standard A	0.500	25	0.09490
G	Standard A	0.250	25	0.04745
H	Standard A	0.100	25	0.01898
I	Standard A	0.500	25	0.00949
J	Standard A	0.000		0.000

SAMPLE COLLECTION, PRESERVATION AND HANDLING

Per GE45:10.20.96:ML01, Manual Distillation and Manual Colorimetric Determination.

PROCEDURE

Add 2 spatula scoops of calcium hydroxide to a disposal centrifuge tube and transfer the 50 ml of absorber solution to it. Shake it up well. Allow to settle overnight or filter on nylon 0.45 µ membrane filters. This is to remove carbon dioxide which may cause air bubbles in the system.

Turn on the system unit (QuickChem AE) and data manager (IBM PS computer). Install the Cyanide Reaction Module. Set up the Cyanide Method and Reaction Module per data in Appendix C before the first run. Subsequent use will require only downloading the existing method (Directions below).

Fill water containers with fresh water. Put the reagent lines into a 1-L beaker filled with DI water. Start the reagent pump and clamp down the tube cartridges. When all air bubbles have passed the photometer head, down-load the Cyanide method from the Analysis-Select Menu option.

When the Edit menu comes up, move to the right to the Programs option. Select Load Calibration. Select the latest calibration or calibrate at this time and tap Enter. The Edit menu will return. Select Identification. Key sample ID information into the identification field and enter any dilution factors into Dilution. (Sample volume of 250 mL and absorber final volume of 50 mL are assumed and programmed into the report generator program. Reduced sample volumes, due to known high level samples, must be taken into account: divide 250 by the actual sample volume and enter the result as the dilution factor.)

Cyanide concentrations for solid samples are calculated on a mg/kg dry weight basis from percent solids determinations conducted separately.

Save the template: Escape to the Edit menu and move left to the File menu. Select Write Template (Method). Name it as a pseudo date: 95062301, for example. When you tap Enter, the template will be

written to disk. Print out a copy to use as a benchsheet via the Export selection of the File menu.

Transfer clear absorber solution to analyzer tubes in the order given in the template. Place the reagent lines in the proper reagents and cover the necks of the bottles with parafilm: Refer to the Method Diagram in Appendix C for the tube colors which correlate to the chemicals.

Place vials of 1 mg/L standard in the tray at positions C and 14. Put a vial of carrier in position 10. Place the tray on the right end of the sampler rack.

Submit the sample tray and observe results intermittently in the run-time-partition while continuing other duties.

When the run is finished, transfer the carrier and reagent suction lines to the wash beaker. Run water through all lines for 4 min. then pump the lines dry (4 min.). Release the tube cartridges and shut down the pump. Meanwhile, print the post-run report, check PIF's, LCS/LCSD and MS/MSD recoveries.

QUALITY CONTROL/QUALITY ASSURANCE

A calibration curve with (10) points (0.00 to 5 mg/L, per Table I) is analyzed at least quarterly. Linear regression must provide a correlation coefficient ≥ 0.995 in each segment. It is verified by running fresh standards, per Table I, which must be recovered 95.0 to 105.0%, quarterly. If the calibration fails to meet acceptance criteria the cause should be investigated and corrected. If this effort fails, recalibrate.

An initial calibration verification standard (ICVS, 1.0 mg/L) is analyzed each time the calibration is loaded. A blank and a continuing calibration verification standard (CCVS, 1.0 mg/L) are run at the beginning of each tray, after every 10 samples or QC items, and at the end of each tray.

One MS/MSD pair, one LCS/LCSD pair, and one distilled method blank are analyzed for each batch of 20 samples or fraction thereof. LCS/LCSD recovery and method blank must meet the CQAP criteria for sample data to be reportable.

PREVENTIVE MAINTENANCE

Sample pump tubes (2 greens) need to be replaced monthly. Pump tubes on the board need to be replaced at least bimonthly, more often when run frequency exceeds 2 per week.

Valve flares need to be replaced annually.

Wash up chemical spills promptly.

TROUBLESHOOTING

Negative peaks for the instrument blank and low standards are usually due to analyte in the carrier. Replace with fresh and protect from contamination with the analyte.

Slight plugging of the chemical and carrier suction lines and any of the T's on the board will cause timing variations and/or low response. When these symptoms are present, disassemble and inspect the same from the suction weights to the drain. Clear obvious debris with a syringe needle or the drill bit supplied in the Lachat parts kit.

SAFETY

Exercise good common sense when handling chemicals and samples. Wear rubber gloves, a labcoat and safety glasses.

Work in a hood when making Py-Bar reagent.

REFERENCES

15.1 Savannah Laboratories Comprehensive Quality Assurance Plan

15.2 Methods for Chemical Analysis of Water and Wastes: U.S. EPA Office of Research and Development: Cincinnati, OH, March 1983 EPA 600/4-79-020 Revised march 1983.

15.3 Test Methods for Evaluating Solid Waste, SW-846, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., Update I, II and III, September 1994.

15.4 Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition, APHA; Washington, D.C., 1992.

15.5 Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office, Washington D.C., July 1, 1993.

APPENDIX A, Example run template:

```
+Operator:BB
+Comment1:CN, 9012, 0.010 to 5.0
+Comment2:
+Comment3:
+Comment4:
+01,LCS G-III-20- 7 4.0    ,1,  1.00,  1.0000,  4.0000,  1.0000,
+02,LCS G-III-20- 7 4.0    ,1,  1.00,  1.0000,  4.0000,  1.0000,
+03,                ,1,  1.00,  1.0000,  0.0000,  1.0000,      +04,+ Matrix Spike        ,1,  1.00,  1.0000,
0.5000,  1.0000,
+05,+ Matrix Spike Duplicate,1,  1.00,  1.0000,  0.5000,  1.0000,
+06,
+07,
+08,..              ,1,  1.00,  1.0000,  0.0000,  1.0000,
+09,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+10,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+11,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+12,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+13,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+14,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+15,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+16,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+17,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+18,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+19,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+20,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+21,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+22,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+23,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+24,Unspike 2           ,1,  1.00,  1.0000,  0.0000,  1.0000,
+25,+ Matrix Spike        ,1,  1.00,  1.0000,  0.5000,  1.0000,
+26,+ Matrix Spike Duplicate,1,  1.00,  1.0000,  0.5000,  1.0000,
+27,SLES DI Water         ,1,  1.00,  1.0000,  0.0000,  1.0000,
+28,+ Reag Watr Spike     ,1,  1.00,  1.0000,  0.5000,  1.0000,
+29,+ Reag Watr Spike Duplic,1,  1.00,  1.0000,  0.5000,  1.0000,
+30,..              ,1,  1.00,  1.0000,  0.0000,  1.0000,
+31,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+32,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+33,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+34,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+35,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+36,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+37,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+38,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+39,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+40,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+41,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
+42,                ,1,  1.00,  1.0000,  0.0000,  1.0000,
```

APPENDIX B:

Barbituric Acid: Acros 18092-5000

Calcium Hydroxide: Mallinkrodt 4195-500

Chloramine-T: Fisher O1779-250

Hydrochloric Acid: Baker 9530-33

Potassium Cyanide: Fisher P226-500

Pyridine: Fisher P368-500

Sodium Arsenite; Fisher S225-100

Sodium Hydroxide: E. Merck SX0593-3

APPENDIX C:

QuickChem AE Method Parameters
Cyanide

Software:

Detector Boundary Segments: A - F and F - I

Signal Processing: Direct Absorbance, ADC = -1

Auto Dilution: Not Used

Data Window: Top Scale: 0.50 Bottom Scale: 0.0

Mode; Manual
Cycle Period: 61 s
Transition to LOAD: 20 s after start of cycle
Transition to INJECT: 30 s after start of LOAD

Lead baseline start; 27 s after inject
Lead baseline width: 2 s
Peak Start: 29 s after inject
Peak Width: 43 s
Trailing Baseline Start 72 s after inject
Trailing Baseline Width 2 s

Standards Protocol: a b c d e f f g h i i

Unit Timing;
Cycle Period; 61 s
Probe in Sample: 35.5 s
Mode: Automatic

Pump to standby after no activity for 600 s
Analysis start waits 20 s after tray submission to assure flow stability.

Board Configuration:
Sample Loop: 150 cm
Wave Length: 570 nM
Heat: 650 + 175 cm @ 60°C
Pump Speed: 37

Tube Colors:
Carrier: Orange
Buffer: Orange
Chloramine-T: Orange
Py-Bar: Gray

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager
Date: 5/1/98

MERCURY: VARIAN SPECTRA AA 20

1.0 SCOPE AND APPLICATION

This method describes the cold-vapor atomic absorption procedure for the determination of mercury in liquids, groundwaters, soils and sludges.

The practical quantitation limit for this method is 0.2 μg/L in fresh waters or in saline waters, and 0.010 mg/kg in solid matrices.

2.0 SUMMARY

This method is based on the absorption of characteristic radiation at 253.7 nm by mercury vapor. After digestion, the mercury is reduced and aerated from solution in a mixing coil. The mixture passes through a gas/liquid separator and then vapor passes through a flow cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. This procedure is adapted from Methods 7470 and 7471 from SW-846 (2).

3.0 INTERFERENCES

Potassium permanganate is added to eliminate the possibility of sulfide interference and digested organic.

Chlorine is known to interfere. Because chlorides are oxidized to free chlorine during the oxidation step, addition of extra permanganate may be necessary for chloride-containing samples.

During digestion, sample bottles are left open so free chlorine can escape. Also, free chlorine can be reduced by using an excess of hydroxylamine sulfate reagent (25 mL).

4.0 APPARATUS AND MATERIALS

Varian Spectra AA 20: This provides a mount for the absorption cell and mercury lamp.

Mercury hollow cathode lamp

Absorption cell: The cell is 17½ cm long with quartz end windows.

VGA 76 - Vapor generation accessory

Mixing coil

Pump tubing: Tygon tubing is used to pass the sample from the test tube through the mixing coil into the gas/liquid separator.

Volumetric glassware

Printer

5.0 REAGENTS AND STANDARDS

DI water: DI water is monitored for impurities. Conductivity is checked daily and must be < 1 μmho in order to be used.

Nitric acid, concentrated: reagent grade

Sulfuric aid, concentrated: reagent grade

Hydrochloric acid, concentrated: reagent grade

Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated HCL to one volume of concentrated HNO_3.

Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 50 g of $KMnO_4$ in 1000 mL of DI water.

Sodium chloride-hydroxylamine sulfate solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine sulfate in DI water in a 1-L volumetric flask and dilute to volume.

Stannous chloride: Add 100 g of stannous chloride in DI water in a 500-mL volumetric flask, add 125 mL hydrochloric acid, concentrated, and dilute to volume.

Potassium persulfate, 5% solution (w/v): Dissolve 50 g potassium persulfate in 1000 mL of DI water.

Commercial stock standard, (Baker) mercury, 1000 ppm

Mercury intermediate stock standard, 10 ppm: Add 1 mL stock standard, 1000 ppm, and 2.5 mL nitric acid to some DI water in a 100-mL volumetric flask. Dilute to volume with DI water.

Mercury intermediate working standard 0.05 ppm: Add 5 mL intermediate stock standard and 2.5 mL nitric acid to a 1-L volumetric flask and dilute to volume.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Collect a representative sample in a plastic red-dot (nitric acid) bottle. Check pH in laboratory and if necessary, acidify the sample to pH < 2 with nitric acid.

Collect soils in a plastic bottle and cool samples to 4° C.

Holding time is 28 days from date of sampling.

Holding time for CLP (3) samples is 26 days from date of sample receipt.

7.0 PROCEDURE

7.1 Calibration Standard Preparation

Transfer 0.2-, 0.4-, 1.0-, and 5.0-mL portions of intermediate working standard to a series of 125-mL glass bottles. Add DI water from a graduated cylinder to each bottle to make a final volume of 50 mL. This results in working standard concentrations of 0.2, 0.4, 1.0, and 5.0 μg/L

mercury. Shake well and add 2.5 mL of concentrated H_2SO_4, 1.25 mL of concentrated HNO_3, and 7.5 mL of $KMnO_4$ solution and let stand at least 15 min. Add 4 mL of potassium persulfate and heat for 2 h in a water bath at 95° C. Cool and add 3 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. When the solution has been decolorized (excess permanganate reduced), place in test tubes onto autosampler.

7.2 **Sample Preparation**

Liquid samples: Add 50 mL of sample or a dilution brought to 50 mL to a 125-mL glass bottle. Add 1.25 mL HNO_3, 2.5 mL H_2SO_4, and 7.5 mL of $KMnO_4$ solution to each sample. Shake well after each addition. Be sure the purple color persists for at least 15 min. If not, add up to three times more $KMnO_4$ solution. Then add 4 mL of potassium persulfate to each sample, shake well, and place the samples in a water bath at 95° C for 2 h. Remove and allow the samples to cool. Add 3 mL of hydroxylamine sulfate solution to each bottle to neutralize excess $KMnO_4$. Follow the analysis procedure below.

Soil Samples: Weigh between 0.80 and 1.00 g wet weight of sample into a 125-mL glass bottle. Add 2.5 mL DI water and 2.5 mL aqua regia. Heat for 2 min in waterbath at 95° C, cool, then add 25 mL DI water and 7.5 mL $KMnO_4$ solution to sample. Mix and heat for 30 min at 95° C. Cool and add 3 mL sodium chloride-hydroxylamine sulfate solution to reduce excess $KMnO_4$. Add 27.5 mL DI water and shake well.

Saltwater: Use 100 mL of sample in order to get a lower detection limit of 0.1 µg/L. Concentrate the sample to 50 mL by heating on a hot place in a Teflon beaker. Add 4 mL H_2SO_4, 2.5 mL HNO_3, and 0.5 mL of $KMnO_4$ solution and mix well. If the pink color disappears after 15 min, add additional amounts of $KMnO_4$ solution until the pink color persists. Usually, about 7 mL is required. Prepare two blanks in the same manner. **Do not** heat in water bath. Then add 1 mL hydroxylamine sulfate solution to neutralize excess $KMnO_4$. Follow the analysis procedure below.

Fish and Crustaceans: Weigh between 0.20 and 0.30 g of the sample and place into a 125-mL glass bottle. Add 2 mL H_2SO_4 and 0.5 mL HNO_3 to each sample and digest in the waterbath for 30 min at 80° C, or until completely dissolved. After samples are cooled, add 7.5 mL of $KMnO_4$, and put samples back into water bath for an additional 90 min at 30° C. Remove and cool. Add 3 mL hydroxylamine sulfate solution to neutralize excess $KMnO_4$. Blanks and QC-check standards should be treated identically. Use 0.20 to 0.40 g of EPA "Trace Metals in Fish" standard for the QC-check standard. Fish are calculated on an "as is" basis. Follow the analysis procedure below.

Prepare one blank and two QC-check standards for each 20 samples. These QC check standards are to be used as ICV/CCV and liquid lab control standards. The blank will be used as ICB/CCB and preparation blank. The blanks are prepared using 50 mL of DI water and all the reagents. The QC-check standards are prepared by using 0.5 mL of EPA QC-check sample concentrate (or equivalent) diluted to a final volume of 50 mL. The QC-check standard will serve as an initial calibration standard and continuing calibration verification standard. Treat blanks and QC-check standards as samples.

7.3 **Analysis**

CAUTION: Mercury is toxic. Insure that the exit line from the spectrophotometer is led into a absorbing media in an exhaust hood or adjacent to a vent.

Fill test tubes with digested samples and label. Fill standard tubes, calibration blank tubes and rinse bottle with appropriate solutions and label. Fill one of the VGA 76 250-mL reservoirs with DI water and the other with the stannous chloride solution. Arrange samples on autosampler tray and assign places on computer. Set instrument parameters to manufacturing specifications. Align mercury lamp and flowcell. Turn on the BGA 76. Recall program on computer. Then

run calibration standards and print out the calibration curve. If curve is satisfactory (i.e., correlation coefficient ≥ 0.995), proceed in running the tray.

Usual tray setup: ICV, ICB. Then add up to 10 samples, CCV, CCB. 10 more samples. CCV, CCB, etc. At the end of the tray, run CCV, and CCB.

ICV = initial calibration verification.
ICB = initial calibration blank.
CCV = continuing calibration verification
CCB = continuing calibration blank.

These standards are defined above, under sample preparation.

The computer will calculate and print out results in μg/L as the samples are analyzed.

All EPTOX extracts, all samples that suffer from matrix interferences, and all samples analyzed as part of a delisting petition are analyzed by the method of standard addition. The standard addition method used is a single-point addition, adding 1 mL of 0.05 ppm mercury working standard to 50 mL of sample, for an added concentration of 1 μg/L.

All dilution or concentration factors must be taken into account. All soils and sludges must be appropriately qualified (*e.g.*, 0.19 mg/kg dw).

7.4 Calculations

Liquids -- The instrument readout is in μg/L. The normal reporting units are mg/L, therefore, the answer must be corrected for units and any dilution or concentration procedure that was used on the sample.

Example: Consider an instrument readout of 0.452 μg/L Hg.

1. If 50 mL of sample is digested and the final volume of the digested sample is 50 mL, the result would only need to be corrected for units: 0.452 μg/L * (1 mg/1000 ug). The Mercury result would be 0.00045 mg/L.

2. If 5 mL of sample is digested and the final volume is 50 mL, we have diluted the sample by a factor of 10. In this case, correct the instrument readout by a factor of ten before correcting for units. The reported answer would be 0.0045 mg/L.

Solids -- If the sample is a solid, we must consider both the weight of the sample digested and the final volume to which the sample is diluted. The instrument generated units of μg/L must be converted to mg/kg as is or mg/kg dry weight basis.

$$\frac{mg}{kg}\,dw = \frac{(X\ \mu g/L) * (V\ mL) * (1\ L/1000\ mL) * (1\ \mu g/g)/(1\ mg/kg)}{W(S)}$$

where
X = instrument readout in μg/L
V = volume of digested sample
W = wet weight of the sample in g
S = % solids (expressed as decimal equivalent (i.e., 85% solids is 0.85)

To report data as mg/kg as is, simply omit the % solids from the calculation.

8.0 QUALITY CONTROL/QUALITY ASSURANCE

The instrument is calibrated using a five-point calibration curve. The levels are as follows: 0.2, 0.4, 1.0, 3.0, and 5.0 µg/L. These standards are digested following the sample preparation procedure.

Immediately following calibration, an initial calibration verification standard (ICV) from an independent source must be analyzed. The ICV is prepared following the sample preparation procedure. Results must be within 15% of the true value. If not, the analysis is terminated and hte problem corrected before proceeding.

Calibration verification standards (CCV) should be analyzed after every 10 samples and at the end of each run and must be within 20% of the true value. Samples must be rerun that are not bracketed by calibration verification standards meeting this criterion.

Lab control samples should be processed in duplicate for each batch or for every 20 samples, whichever is more frequent. This will allow the determination of accuracy and precision. If the lab control samples do not fall within 80-120% of the true values of the metal of interest, the batch must be redigested and reanalyzed.

A calibration blank (ICB) must be analyzed inmediately following the ICV and after each calibration verification standard (CCB). The ICB and CCB must be below the practical quantitation limit.

For soil samples, digest and analyze an NIST standard reference material (NBS 1646 estuarine sediment) in duplicate for every 20 samples. the determined value must fall within 70-130% of the true value.

A matrix spike sample and a matrix spike duplicate sample are analyzed for every 20 samples. The matrix spike is at 1 µg/L mercury.

Dilute samples if they are higher in concentration than the highest standard.

9.0 REFERENCES

1. *Methods for Chemical Analysis of Water and Wastes*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983.

2. *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

3. *US EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, Multi-media, Multi-concentration Document Number ILM01.0.*

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

MERCURY ANALYSIS: LEEMAN PS200

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure to determine the concentration of mercury by cold vapor atomic absorption spectrophotometry (CVAA). This method contains the analytical procedures for determination of mercury in liquids, surface and groundwaters, soils, sediments, sludges, wastes and leachates (EP or TCLP) after digestion. The digestion procedures for mercury are contained in SL SOPs ME29.

1.2 The reporting limit (RL) and the accuracy and precision criteria are listed in Section 5 of the current revisions of the Savannah Labs' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF THE METHOD

2.1 This method is based on the absorption of characteristic radiation at 253.7 nm by mercury vapor. After a digestion, the mercury is reduced by the addition of stannous chloride and aerated from solution in a mixing coil. The mixture passes through a gas/liquid separator and the vapor is then passed through a drying tube. The vapor is then passed through a flow cell positioned in the light path of an atomic absorption spectrophotometer. Mercury concentration is then measured as a function of absorbance.

2.2 This method is based on the guidance provided in SW-846 methods 7470A, 7471A, and EPA method 245.1 (Drinking Water version).

3.0 SAFETY

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

3.2 Acid spill kits must be available. These kits must be located in a highly accessible area of the lab. Each lab must have access to a properly working shower.

3.3 The standards and reagents used in this method should be treated as potential hazards. Lab coats, gloves, safety glasses and other protective equipment must be used when preparing samples, standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are available to the analyst/chemist. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions. Each person should read and understand these sheets for all standards and reagents used before beginning this procedure.

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate the possibility of interference from sulfide and certain organic compounds.

4.2 Chlorine is known to interfere. Addition of extra potassium permanganate may be needed during the digestion of chloride containing samples. Also, the samples are not capped tightly during digestion so that excess chlorine can escape.

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain mercury. Reagent blanks (method blanks) must be analyzed as a check on contamination due to sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

5.1 Aqueous samples and TCLP/EP-TOX Leachate

5.1.1 Liquid samples are collected in 250-mL plastic or glass containers. The samples are preserved with HNO3 to a pH <2. The samples must then be stored at 4C (less than 6C but not frozen) until the samples are prepared for analysis. Samples must be digested and analyzed within 28 days of collection.

5.1.2 Samples for dissolved mercury 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/Waste Samples

5.2.1 Soil and sediment samples are collected in 250-mL or 500-mL plastic or glass containers. The samples are iced at the time of collection and stored at 4C (less than 6C but not frozen) until the time of digestion and analysis. Samples must be digested and analyzed within 28 days of collection.

6.0 APPARATUS AND MATERIALS

6.1 Leeman PS200 or other suitable automated mercury analyzer with data system and printer

6.2 Nitrogen gas supply and appropriate fittings

6.3 Pump tubing of appropriate sizes for use on the PS200

6.4 Volumetric glassware for making standards and reagents

6.5 Test tubes of the two sizes to fit the PS200 autosampler

7.0 REAGENTS

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with SL SOP AN35.

7.2 Nitric Acid (HNO3), concentrated-reagent grade

7.3 Hydrochloric Acid (HCl), concentrated-reagent grade

7.4 Rinse Water, 5% HCl-1%HNO3 - to a clean 2-L bottle add 1-L of reagent water. Carefully add 100-mL of concentrated hydrochloric acid. Carefully add 20 mL of concentrated nitric acid. Dilute to a final volume of 2-L. Other volumes may be utilized providing the reagent proportions remain the same.

7.5 Stannous chloride (SnCl2.2H2O) - reagent grade, suitable for mercury determination

7.5.1 Stannous chloride (SnCl2.2H2O) solution - to a clean 2-L volumetric flask add 100-g of stannous chloride. Add approximately 400-mL of reagent water. Carefully add 500-mL of concentrated hydrochloric acid. Add a stirring bar and stir on a stir plate until the stannous chloride is dissolved. Remove the stirring bar and dilute to volume with reagent water.

7.6 Magnesium perchlorate (Mg(ClO4)2) - used as a drying agent in the drying tube. The magnesium perchlorate should be as coarse as possible

8.0 STANDARD PREPARATION

For the preparation of standards see the appropriate section of SL SOP ME29. Note that the standards are digested in the same manner as the field samples.

9.0 SAMPLE PREPARATION

For the preparation of samples see the appropriate section of SL SOP ME29.

10.0 ANALYSIS PROCEDURE

10.1 Initial startup of the instrument

10.1.1 Before analysis begins inspect the system (pump tubes, mixing coil, gas/liquid separator) to see if any parts need to be cleaned or replaced.

10.1.2 Replace the drying tube with a freshly packed drying tube, making sure that the magnesium perchlorate is not packed too tightly. The vapors must be able to pass freely through the drying tube.

10.1.3 Fill the rinse tank with rinse water.

10.1.4 If the lamp is not already on and warmed up perform a cold-start using the COLDSTRT macro. The lamp must warm up for a minimum of 2 hours.

10.1.5 If the lamp is already on and warmed up, perform a warm-start using the WARMSTRT macro. This will give the instrument time to "exercise" the pump tubes. Allow a minimum of 20 minutes of pump time for the pump tubes to break in each day.

10.1.6 Check the aperture. If the aperture is not close to 0 (+/- 100) adjust the appropriate set screw. If the aperture must be adjusted, allow at least 10 minutes for the cell to warm up after replacing the cover.

10.1.7 Fill the stannous chloride reagent bottle with stannous chloride solution. Switch the reagent line from rinse to the stannous chloride reagent bottle.

10.2 Autosampler setup

10.2.1 Fill the standard tubes with the appropriate standards for the protocol being followed. Refer to ME26 or ME29 for the prep of these standards.

10.2.2 Fill the labeled sample test tubes with the samples and calibration verification standards in the applicable order. An example order is as follows:

ICV - Initial Calibration Verification Standard
ICB - Initial Calibration Blank
10 SAMPLES
CCV – Continuing Calibration Verification Standard
CCB - Continuing Calibration Blank
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
:
:
CCV
CCB

It is appropriate to use the liquid laboratory control samples as the CCV. The specific ID for the LCS is used to identify the CCV (i.e., 1209T-1, 1209T-2). The CCVs will bracket the samples which they were prepped with.

The preparation blank will be analyzed first. The LCS will follow immediately after the preparation blank. The samples, matrix spikes and duplicates will then follow with a maximum of 10 analyses between CCVs/CCBs. All samples and control samples will be labeled with the corresponding batch ID.

10.2.3 Enter the sample/QC IDs into the autosampler table giving each rack a unique name.

10.2.4 Load the rack(s) onto the autosampler.

10.3 Calibration of the mercury analyzer.

10.3.1 Call up the required protocol. Open a new data folder. Enter the operator ID (e.g., J.Smith).

10.3.2 Go to CALIBRATION, RESET, and reset the calibration for a new calibration

10.3.3 Go to CALIBRATION, STANDARDS, and insure that calibration standards are entered at the proper concentrations.

10.3.4 Analyze the standards, beginning with standard 1 (Blank), proceeding from lowest to highest concentration.

10.3.5 When all calibration standards have been analyzed, go to CALIBRATION, LINE CALIBRATION. If calibration is within acceptable limits (correlation > 0.995) accept the linear calibration and print the calibration curve.

10.4 Sample analysis

10.4.1 Go to AUTOSAMPLER, SETUP. Enter the Rack ID (s) and the cup numbers to be analyzed.

10.4.2 Press the SAMPLE (F8) key to begin the analysis run.

10.5 If the concentration of a sample is above the calibration range of the Hg analyzer, the sample digestate must be diluted and reanalyzed. The amount of digestate needed to prepare the desired dilution is determined from the following equation.

$$V_{digest} = (Vf_{vol})/DF$$

where

V_{digest} = volume of sample digestate used to make the dilution (mL)
Vf_{vol} = final volume of diluted sample (mL)
DF = dilution factor

10.5.1 The dilution factor is calculated as follows:

$$DF = Vf_{vol}/V_{digest}$$

where

V_{digest} = volume of sample digestate used to make the dilution (mL)
Vf_{vol} = final volume of diluted sample (mL)
DF = dilution factor

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Aqueous and Leachate Samples

The concentration of mercury in liquid samples is calculated as follows:
(Note that results may also be reported in mg/l. Results in mg/l are reported by dividing the result below by 1000).

$$C_{sample} = C_{curve} \times DF$$

where

C_{sample} = concentration of sample (ug/L)
C_{curve} = concentration from curve (ug/L)
DF = dilution factor

The reporting limit (RL) is calculated as follows:
(Note that results may also be reported in mg/l. Results in mg/l are reported by dividing the result below by 1000).

$$RL_{sample} = RL_{qap} \times DF$$

where

RL_{sample} = reporting limit of sample (ug/L)
RL_{qap} = reporting limit from Table 5 of CQAP (ug/L)
DF = dilution factor

11.2 Soil/Solid Samples

The concentration of mercury in soil and solid samples is calculated as follows:

$$C_{sample} = C_{curve} \times [(F)/(W \times solids)] \times DF \times 1mg/1000ug$$

where

C_{sample} = concentration of sample (mg/kg dw)
C_{curve} = concentration of digest from curve (ug/L)
F = final volume of digest (L)
W = weight of sample digested (kg)
solids = (percent solids)/100
DF = dilution factor

The reporting limit (RL) for soil/solid samples is calculated as follows:

$$RL_{sample} = [RL_{qap}/(W \times solids)] \times DF$$

where

RL_{sample} = reporting limit of sample (mg/kg dw)
RL_{qap} = reporting limit from Table 5 of CQAP (mg/kg)
W = weight of sample digested (kg)
solids = (percent solids)/100
DF = dilution factor

The RL is based on a 1-gram sample digested to a final volume of 50-mL.

11.3 The theoretical concentration (CT) of a spiked sample is calculated:

$$C_T = (C_s \times V_s)/(V_{sample})$$

where

C_s = concentration of the matrix spiking solution (mg/L)
V_s = volume of the matrix spiking solution added to the sample (mL)
V_{sample} = volume of the sample spiked (mL)

The theoretical concentration (CT) of a soil matrix spike is calculated:

$$CT = (C_s \times V_s \times (1\text{-}L/1000\text{-}mL))/(W \times solids)$$

where

C_s = concentration of the matrix spiking solution (mg/L)
V_s = volume of the matrix spiking solution added to the sample (ml)
W = weight of sample digested (kg)
solids = (percent solids)/100

11.3.1 The concentration of mercury in the lab control and matrix spike samples is determined as in Section 11.1.1. The concentration is compared to the theoretical spike concentration and the percent recovery is calculated.

The percent recovery is calculated:

$$\%REC = [(C_{MS} - C_{sample})/(C_T)] \times 100$$

where

C_{MS} = concentration of the spiked sample (mg/L or mg/kg dw)
C_{sample} = concentration of the unspiked sample (mg/L or mg/kg dw)
C_T = theoretical concentration of the spike (mg/L or mg/kg dw)
(Assume C_{sample} = 0 for the LCS/LCSD)

11.3.2 The relative percent difference is calculated:

$$\%RPD = |(REC_{MS} - REC_{MSD})/((REC_{MS} + REC_{MSD})/2)| \times 100$$

where

REC_{MS} = percent recovery of the MS (or LCS)
REC_{MSD} = percent recovery of the MSD (or LCSD)

12.0 QUALITY CONTROL/QUALITY ASSURANCE

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..

13.0 PREVENTATIVE MAINTENANCE

13.1 Pump tubing: Inspect daily and replace as needed.

13.2 Standard Autosampler Cups: Clean daily and replace as needed.

13.3 Drying Tube: Repack daily, or more often if needed.

13.4 Mixing Coil: Inspect weekly, clean and replace as needed.

13.5 Sample Probe: Inspect monthly, clean and replace as needed.

13.6 Mercury Lamp: Clean or replace as needed.

14.0 TROUBLESHOOTING

No items for this section in current revision of SOP.

15.0 REFERENCES

(1) Savannah Laboratories' *Comprehensive Quality Assurance Plan* and Savannah Laboratories *Corporate Quality Assurance Plan*, current revisions

(2) *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986 (Update III).

(3) *Methods for Analysis of Water and Waste*; U.S. EPA Office of Research and Development: Cincinnati, OH, March 1983.

Method Summary - HG analysis

HOLD/STORAGE

Container	Minimum 250mL plastic or glass bottle with a plastic or Teflon-lined lid.
Preservation	HNO3 to pH <2 in the field. If dissolved mercury is required, filter the samples before preservation.
Storage	Liquids and solids should be stored at 4C +/- 2C from collection until preparation.
Hold Time	Samples may be held for up to 28 days from the time of collection.

SAMPLE PREPARATION

Samples should be prepared in accordance with ME26 or ME29.

ANALYTICAL SEQUENCE

Instrument Startup	Turn on the mercury analyzer according to the instrument manufacturer's recommendations. Allow the mercury lamp proper warm-up time. Inspect and change pump tubes and drying tubes as needed. Check and align lamp and cell according to the instrument manufacturers recommendations.
Initial Calibration	Beginning with the blank, calibrate with the blank and 5 standards. One standard must be at the PQL.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the analysis run. The ICV solution must come from a source other than the calibration source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with a concentration at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run. The CCV and ICV may be the same solution. Analyze a calibration blank after every CCV.
Detection Limit Check Solution	At the beginning of the analysis run, verify the accuracy at the PQL by analyzing a standard with a concentration at the required PQL/CRDL.
Post Digestion Spikes	At a minimum of once per analytical batch, verify the absence of matrix interference by analyzing a post digestion spike.

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QC CRITERIA

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 blank and 5 standards Correlation > 0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within +/- 10% 245.1 = within +/- 5%	Recalibrate
Continuing Calibration Verification Standards (CCV)	At the beginning and end of the analysis and every 10 samples.	SW846 = within +/- 20% 245.1 = within +/- 10%	Terminate the analysis, correct 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 PQL/CRDL.	SW846 = terminate the analysis, correct the problem and reanalyze the previous 10 samples. s.
PQL/CRA standard	After every calibration but not before the ICV.	Absolute limits = +/- 50% of true, in-house limits should be established.	Recalibrate.
Laboratory control sample (LCS)	One per batch of twenty samples or less	In SL Quality Assurance Plan	Redigest and reanalyze batch
Preparation Blank - SW846	One per batch of twenty samples or less	|result| < PQL.	Redigest and reanalyze batch
MS/MSD - SW846	One set per batch of twenty samples or less	%Rec = 80 - 120% %RPD = < 20%	Flag and report data
MS - 245.1	MS added to a minimum of 10% of samples	%Rec = 70 - 130%	Flag and report data
Post Digestion Spikes	One per batch of twenty samples or less	%Rec = 85 - 115%	Check for interference source and reanalyze samples or analyze samples by MSA.
Detection Limit Check Solution	At the beginning of the analysis but not before the ICV	%Rec = 50 - 150%	Terminate the analysis, fix the problem, reanalyze the solution.

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager Date: Jan 20, 1998

MERCURY PREPARATION: LEEMAN AP200

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the automated digestion procedure for mercury analysis by cold vapor atomic absorption spectrophotometry (CVAA). This method contains the digestion procedure for determination of mercury in liquids, surface and groundwaters, wastes and leachates (EP or TCLP). The analytical procedure for mercury is found in SL SOP ME28:*Mercury Analysis-Leeman PS200.*

1.2 The reporting limit (RL) and the accuracy and precision criteria are listed in Section 5 of the current revisions of the Savannah Labs' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF THE METHOD

2.1 A known volume of sample is transferred to a test tube. The sample is placed into an autosampler rack and reagents are automatically added to each sample. The samples, along with calibration and QC standards, are digested in diluted acid, potassium permanganate-potassium persulfate solutions and oxidized for 2 hours at 95°C. After the digestion is complete, the samples are analyzed using SOP ME28.

2.2 This method is based on the guidance in methods 7470A and method 245.1.

3.0 SAFETY

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

3.2 Acid spill kit must be available. These kits must be located in a highly accessible area of the lab. Each lab must have access to a properly working shower.

3.3 The standards and reagents used in this method should be treated as potential hazards. Lab coats, gloves, safety glasses, face-shields, and other protective equipment should be used when preparing samples, standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are available to the analyst/chemist. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions. Each person must read and understand these sheets for all standards and reagents used before beginning this procedure.

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate the possibility of interference from sulfide and certain organic compounds.

4.2 Chlorine is known to interfere. Addition of extra potassium permanganate may be needed during the digestion of chloride containing samples. Also, the samples are not covered tightly during digestion so that excess chlorine can escape.

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain mercury. Reagent blanks (method blanks) must be analyzed as a check on contamination due to sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

5.1 Aqueous and TCLP/EP-TOX Leachate Samples.

Liquid samples are collected in 250mL plastic or glass containers. The samples are preserved with HNO_3 to a pH <2. The samples must then be stored at 4C (less than 6C but not frozen) until the samples are prepared for analysis. Samples must be digested and analyzed within 28 days of collection.

Samples for dissolved mercury 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.

6.0 APPARATUS AND MATERIALS

6.1 Leeman AP200 or other suitable automated mercury digestion unit with data system and printer

6.2 Nitrogen gas supply and appropriate fittings

6.3 Volumetric glassware for making standards and reagents

6.5 Test tubes of the two sizes to fit the AP200 autosampler

7.0 REAGENTS

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with SL SOP AN35.

7.2 Nitric Acid (HNO_3), concentrated-reagent grade

7.3 Hydrochloric Acid (HCl), concentrated-reagent grade

7.4 Sulfuric Acid (H_2SO_4), concentrated-reagent grade

7.5 Potassium Permanganate ($KMnO_4$), mercury free, crystalline

7.6 Potassium Permanganate ($KMnO_4$), 5% solution (w/v): Dissolve 50 g of $KMnO_4$ in 1000mL of reagent water.

7.7 Sodium Chloride (NaCl), ACS, crystalline

7.8 Hydroxylamine sulfate ($(NH_2OH)_2 \cdot H_2SO_4$), crystalline

7.9 Sodium chloride-hydoxylamine sulfate solution - dissolve 120 g NaCl and 120 g $(NH_2OH)_2 \cdot H_2SO_4$ in reagent water in a 1 liter volumetric flask and dilute to volume with reagent water.

7.10 Potassium persulfate, ($K_2S_2O_8$), ACS, crystalline

7.11 Potassium persulfate, 5% solution - In a 1 liter volumetric flask, dissolve 50 g of $K_2S_2O_8$ in reagent water and dilute to volume with reagent water.

7.12 Rinse Water, 5% HCl-1%HNO_3 - to a clean 2L bottle add 1L of reagent water. Carefully add 100mL of concentrated hydrochloric acid. Carefully add 20mL of concentrated nitric acid. Dilute to a final volume of 2L. Other volumes may be utilized providing the reagent proportions remain the same.

8.0 STANDARD PREPARATION

The standards listed in this section must be tracked following the SOP AN41: *Standard Material Traceability*.

8.1 Calibration Standards

8.1 Mercury stock standard, (i.e., Baker), 1000 mg/L Hg.

8.2 Mercury intermediate calibration stock standard, 10 mg/L Hg: Add 20 - 30mL reagent water to a clean 100mL volumetric flask. Add 1mL concentrated nitric acid. Add 1mL stock standard, 1000 mg/L, and dilute to volume with reagent water.

8.3 Mercury working calibration stock standard, 100µg/L Hg: Add 20 - 30mL reagent water to a clean 100mL volumetric flask. Add 1mL concentrated nitric acid. Add 1mL of the mercury intermediate calibration stock standard, 10 mg/L Hg, and dilute to volume with reagent water.

8.4 Mercury calibration standards, 0.00, 0.20, 0.40, 1.0, 3.0, 6.0 µg/L Hg: To six clean 100mL volumetric flasks add 20 - 30mL reagent water. To each of the six flasks add 1mL concentrated nitric acid. Transfer 0.0mL, 0.20mL, 0.40mL, 1.0mL, 3.0mL, and 6.0mL of the working calibration stock standard, 100 mg/L Hg, to the individual flasks. Dilute the flasks to volume with reagent water.

Note: Different concentrations may be used, with supervisor approval, if the standards properly represent the linear range of the instrument.

8.2 Verification Standards

8.2.2 Mercury stock standard, (i.e. Fisher), 1000 mg/L Hg. This standard must be from a source other than the calibration stock standard.

8.2.2 Mercury intermediate secondary stock standard, 10 mg/L Hg: Add 20 – 30mL reagent water to a clean 100mL volumetric flask. Add 1mL concentrated nitric acid. Add 1mL commercial secondary stock standard, 1000 mg/L Hg, and dilute to volume with reagent water.

8.2.3 Mercury working secondary stock standard, 100 µg/L Hg: Add 20 - 30mL reagent water to a clean 100mL volumetric flask. Add 1mL concentrated nitric acid. Add 1mL mercury intermediate secondary stock standard, 10 mg/L Hg, dilute to volume with reagent water.

8.2.4 ICV/CCV/LCS standards, 3.0 µg/L Hg: Add 20 - 30mL reagent water to a clean 100mL volumetric flask. Add 1mL concentrated nitric acid. Add 3mL mercury working secondary stock standard 100 µg/L, and dilute to volume with reagent water.

9.0 STANDARD/SAMPLE PREPARATION

9.1 Load autosampler tray

Rack 1: Calibration standards/QC standards:

Calibration standards: Label six 50mL digestion tubes with the IDs of the calibration standards. Add 24mL of the appropriate calibration standard to the tube. Load these tubes in positions 1 - 6 of rack one. Add an empty waste tube to position 7.

QC Standards, CCVs & CCBs: Label a proportionate number of 50mL digestion tubes with the CCV and CCB ID. The number of samples digested will determine the number of CCVs and CCBs that will be needed. Add 24mL of the CCV standard, 3.0 µg/L, to each of the tubes labeled CCV. Add 24mL of the calibration blank to each of the tubes labeled CCB. Load these in positions 8 - 13 of rack one. Add a tube of the sodium chloride hydroxylamine sulfate solution to position 14.

Rack 2/3: Samples rack

Samples are to be grouped in digestion batches. Each batch will consist of a minimum of a blank and a single laboratory control standard. The digestion batch will contain a maximum of 20 field samples. A matrix spike/matrix spike duplicate will be analyzed with each batch if sample is available. Method 245.1 requires that a minimum of 10% of samples be spiked.

For matrix spikes add 100µL of the mercury working secondary stock standard to 10mL of sample.

For laboratory control standards (LCSW) use the LCS solution, 3.0 µg/L.

Beginning with position 1 of rack 1 add the labeled sample tubes for the digestion batches. An example would be as follows: Position 1 - prep blank, position 2 - LCSW, positions 3 through 24 - twenty samples plus one set of matrix spike/matrix spike duplicate. Add 8mL of the appropriate samples, blank and LCSW to the labeled tubes.

Repeat this sequence until all batches are on the racks ready to digest.

9.2 Instrument startup.

Insert the reagent bottles into their appropriate positions.

Check autosampler rails and clean and oil if necessary (clean and oil at least weekly under normal use).

Check the water bath reservoir and fill as needed.

Turn on gas, computer, and printer.

Perform the following steps to enter the digestion program:

CD\ICP<enter>
APPS<enter>
<F5>(@WAKEUP), repeat 1 - 2 times
Protocol
Get
PRP7470
Utility
user Name
Enter name(e.g., J. Smith)
Menu
Autosampler
Setup
Enter rack name, i.e., 060197S
Enter the sample names into the rack
Menu
Autosampler
Setup
Enter rack name(s)
Setup the calibration standards *(S)*
Setup the calibration verification standards *(C)*, this would include CCVs and CCBs.
Menu
Utility
Diagnostics
Check reagent pressure - should be 5.5 - 6.5 psi
Make a final, visual check of the autosampler and the autosampler file
<F2> Macros
@PRP7470
Follow the instructions on the screen
When the run is complete, replace the reagent bottles with bottles containing reagent water
<F2> Macros
@CLEAN
Repeat 2 - 3 times
<F2> Macros
@ASLEEP
Turn off gas

10.0 ANALYSIS PROCEDURE

The mercury analysis procedure is contained in the SOP ME28.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 If the concentration of a sample is above the calibration range of the Hg analyzer, the sample may be diluted before digestion. The amount of sample needed to prepare the desired dilution is determined from the following equation.

$$V_{sample} = (Vf_{vol})/DF$$

where

V_{sample} = volume of sample used to make the dilution (mL)
Vf_{vol} = final volume of diluted sample (mL)
DF = dilution factor

11.2 The dilution factor is calculated as follows:

$$DF = Vf_{vol}/V_{sample}$$

where

V_{sample} = volume of sample used to make the dilution (mL)
Vf_{vol} = final volume of diluted sample (mL)
DF = dilution factor

11.3 The theoretical concentration (CT) of a spiked sample is calculated:

$$C_T = (C_s \times V_s)/(V_{sample})$$

where

C_s = concentration of the matrix spiking solution (mg/L)
V_s = volume of the matrix spiking solution added to the sample (mL)
V_{sample} = volume of the sample spiked (mL)

12.0 QUALITY CONTROL/QUALITY ASSURANCE

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.

13.0 PREVENTATIVE MAINTENANCE

13.1 Clean and oil autosampler rails at least weekly

13.2 Clean the water bath monthly

13.3 Inspect reagent bottles and caps quarterly. Replace as needed.

13.4 Inspect the tubing at a minimum of six month intervals and replace as needed.

14.0 TROUBLESHOOTING

No items for this section in current revision of SOP.

15.0 REFERENCES

(1) *Savannah Laboratories' Comprehensive Quality Assurance Plan*, and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions

(2) *Test Methods for Evaluating Solid Waste*, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986.

(3) *Methods for Analysis of Water and Waste*; U.S. EPA Office of Research and Development: Cincinnati, OH, March 1983.

ME50:04.01.98:5

Approval Signature:	R. Wayne Robbins
Title: Corporate QA Manager	Date: 4/6/98

DIGESTION PROCEDURES FOR ICP TOTAL METALS AND TOTAL RECOVERABLE METALS IN LIQUID SAMPLES

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used to digest water, drinking water, wastewater, and leachate samples prior to the analysis by ICP (SL SOP ME70). This procedure can be used to digest samples for Trace (axial) ICPs and non-Trace (radial) ICPs.

2.0 SUMMARY OF METHOD

2.1 Total metals and TCLP leachates: A known volume, usually 50mL, of sample is transferred to a digestion vessel. The sample is refluxed with nitric acid at approximately 95C. After sample has digested, as evidenced by a clear, pale yellow color, HCl is added and the sample is brought up to the original volume with reagent water.

2.2 Total recoverable metals: A known volume, usually 50mL, of sample is transferred to a digestion vessel. The sample is refluxed with dilute nitric acid and hydrochloric acid at approximately 95C. After sample has evaporated to approximately 10-20mL, the sample is brought up to the original volume with reagent water.

2.3 Drinking water samples with a turbidity concentration of less than 1 NTU can be analyzed with no digestion if the required quantitation limits can be achieved with no sample preconcentration. The exception to this rule is silver, which requires sample digestion prior to analysis.

2.4 Samples filtered for the determination of dissolved metals do not require digestion if the sample

1) has a low COD(<20mg/L);
2) has a turbidity <1 NTU ;
3) is colorless with no significant odor ; and
4) is of one liquid phase and free of suspended particulates or precipitates after acidification

(40 CFR Part 136 Table 1B-note 4)

2.5 The SOP is based on the guidance in SW-846 Methods 3005A and 3010A and EPA Method 200.7. Note that EPA has promulgated two procedures as EPA 200.7-one for drinking water and one for NPDES. The digestion procedures for both 200.7 methods are performed in the same manner as 3005A for total recoverable metals and 3010A for total metals.

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 samples are digested in strong acid solutions and contain an acid concentration of 10-20% by volume. The analyst must wear protective clothing such as a lab coat or apron. The acids used in this procedure will destroy unprotected clothing. The analyst must wear proper eye protection such as lab glasses or face shield. Acid can be splashed into the eyes from many sources. Gloves must be worn to protect hands from acid burns.

3.3 The acid digestion procedures must be performed under a properly functioning fume hood. The acid fumes from the digestion can cause mild to severe respiratory problems if breathed.

3.4 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.5 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.6 Each analyst should be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in this procedure. These sheets denote the type of hazard that each reagent poses and the safe handling instructions for these compounds.

3.7 Care must be taken when handling the digestion beakers. Before handling a vessel that has been in use, check the temperature to make sure that it is not hot. Make sure that the digestion vessels are placed on a stable platform during and after the digestion. Vibrations from the hood or an unstable platform can cause the beakers to move and possibly to fall and splatter an analyst with a hot acid solution. Hot acids can cause severe skin burns and destroy unprotected clothing.

4.0 INTERFERENCES

Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	ROUTINE CONTAINER	PRESERVATIVE	STORAGE/HOLD TIME
Aqueous (water) and TCLP leachates	500-mL plastic	HNO3 to pH <2	Ambient

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.

The pH of all preserved samples must be checked and documented upon arrival in the lab. If the pH is not within the proper range, additional acid is added to the sample to bring the pH below 2.

- Place a piece of pH paper (wide range or narrow range can be used) on a watch glass or other inert surface.

- Transfer a few drops of the sample to the pH paper and note the color change. If the pH <2, record this in the log and transfer the sample to the storage area.

- If the pH is greater than 2, move the sample under a hood. Add 1:1 nitric acid to the sample in 1mL aliquots, checking the sample pH after each addition, until the pH <2. The volume of 1:1 nitric acid added to the sample should not exceed 1% of the total volume of the sample. For a 500-mL sample, the maximum volume of 1:1 nitric is 5mL. If more acid is required, contact the supervisor for further guidance. An anomaly form (SL SOP CA 85) is initiated for sample that require pH adjustment after arrival into the lab.

NOTE: Samples that are not at pH <2 upon arrival in the lab may contain cyanide or sulfide or may be highly buffered. Working under a hood minimizes the hazard that may be caused by the evolution of hydrogen cyanide or hydrogen sulfide upon acidification of the sample. Be aware that acid/base neutralization reaction may be violent and evolve a good deal of heat.

6.0 APPARATUS AND MATERIALS

6.1 Digestion vessels: Teflon or Pyrex beakers, 150-mL and 250mL, or comparable digestion vessels (250-mL beakers are listed in the preparation steps but 125-mL or 50-mL digestion block digestion vessels can be substituted)

6.2 Watch glasses, "ribbed", to fit over digestion vessels

6.3 Hot plates or digestion block-capable of maintaining a sample temperature of 90-95C. The hot plates or digestion block are calibrated each quarter. The temperature of 50mL of water contained in a digestion vessel at the center of the hot plate or placed in the digestion block is measured and the setting is marked when the temperature of the water reaches 85C (+/- 3C). The temperature of the material in the vessel will rise to approximately 95C +/- 5C when a watch glass is placed on top. (This procedure for calibration is adapted from EPA Methods 200.7 and 200.9, which reference both aqueous and soil/solid digestions)

NOTE: The use of hot plates is listed in the preparation steps but a digestion block may be used if the same general procedures are employed. The hot plate or digestion block settings must be recorded in the maintenance log or other suitable log.

6.4 Volumetric flasks- 100-mL or appropriate volume

6.5 Graduated cylinders-50mL

6.6 Pipettes

6.7 Analytical balance

6.8 Top-loading balance

6.9 Teflon vials-30mL

7.0 REAGENTS

7.1 Reagent water-lab generated deionized water. ASTM Type I or Type II. The conductivity must be checked daily in accordance with SL SOP AN35: *Conductivity Checks for Laboratory Deionized Water.*

7.2 Nitric acid (HNO_3)-reagent grade.

7.3 Hydrochloric acid (HCl)--reagent grade.

8.0 STANDARDS

The preparation of the spiking solutions 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 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

where
Vi = volume of stock standard needed to prepare the spiking solution (mL)
Ci = concentration of stock solution (ug/mL)
Cf = concentration of spiking solution to prepare (ug/mL)
Vf = volume of spiking solution to prepare (mL)

The concentration can be expressed in whatever terms the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for Ci and Cf.

8.2 Preparation of the ICP Matrix Spiking Solutions

8.2.1 ICP Matrix Spiking Solution 1 is a solution purchased from SPEX (catalogue number SPIKE-1). The concentrations of the analytes in this solution are listed on the accompanying certificate of analysis. Store this solution at room temperature. Replace this solution every six months or sooner if needed or required.

8.2.2 Preparation of the ICP Matrix Spiking Solution 2

Add 20-mL to 30-mL of reagent water to a clean100-mL volumetric flask. Add 1-mL of concentrated nitric acid (HNO_3) and 5-mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume.

Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	*Ci(mg/L)*	*Vi(mL)*	*Vf(mL)*	*Cf(mg/L)*
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100

Dilute to a final volume of 100-mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.3 Preparation of the ICP TCLP Matrix Spiking Solutions

8.3.1 ICP TCLP Spiking Solution I

Element	*Ci(mg/L)*	*Vi(mL)*	*Vf(mL)*	*Cf(mg/L)*
Arsenic	1000	5.0	100	50
Barium	1000	5.0		50
Cadmium	1000	1.0		10
Chromium	1000	5.0		50
Lead	1000	5.0		50
Selenium	1000	1.0		10

- Add 1mL of nitric acid and 5mL of hydrochloric acid to a 100mL volumetric flask containing about 50mL of reagent water.
- Add the appropriate volume of each element stock standard to the flask, dilute to volume with reagent water, and mix thoroughly.
- Transfer the spiking solution to a labeled storage container.

8.3.2 ICP TCLP Spiking Solution II

Element	*Ci(mg/L*	*Vi(mL)*	*Vf(mL)*	*Cf(mg/L)*
Silver	1000	5.0	100	50

- Add 1mL of nitric acid and 5mL of hydrochloric acid to a 100mL volumetric flask containing about 50mL of reagent water.
- Add the appropriate volume of each element stock standard to the flask, dilute to volume with reagent water, and mix thoroughly.
- Transfer the spiking solution to a labeled storage container.

9.0 SAMPLE PREPARATION

Unless otherwise requested, groundwater and surface waters will be prepared using the total recoverable metals procedure given in Section 9.1. TCLP samples must be digested for total metals (Section 9.3). Note that there are different spiking solutions for routine and TCLP samples.

The turbidity of drinking water samples can be checked using the procedures in SL SOP BA80:*Turbidity*. If the turbidity of the sample is not checked, the digestion procedure must be performed. If silver must be determined in a drinking water sample, the sample must be digested.

9.1 Total Recoverable Metals: Aqueous Samples

This digestion procedure is used for the preparation of aqueous samples for total recoverable metal determination by ICP (or flame AA). This digestion procedure is not suitable for most analytes which will be determined by graphite furnace atomic absorption (GFAA) because hydrochloric acid (HCl) can cause interferences during furnace atomization. The exception is antimony by GFAA which must be digested using the procedures in this section.

9.1.1 Using a graduated cylinder, transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a clean 250-mL Teflon beaker or other suitable digestion vessel.

NOTE: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample and bring the final digestate volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digestate should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

9.1.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

9.1.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water designated as the laboratory control spike(LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
6010	Liquids (aqueous)	LCS/MS	(Spex) ICP Spike I ICP Spike II

9.1.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spikes sample(MS and MSD).

9.1.5 Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the volume of sample used
- the lot number of the acids used for the digestion
- the lot numbers of the ICP LCS spiking solutions and the ICP matrix spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE:A DIGESTION BATCH CONSISTS OF TWENTY FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. THE BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE(LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE(IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). IF THERE IS NOT SUFFICIENT SAMPLE FOR MS/MSD, THE LCS IS PREPARED IN DUPLICATE(LCSD).

9.1.6 Add 2.5mL of concentrated HCl and 1.0mL of concentrated HNO_3 to each sample. Cover each beaker with a watch glass(a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 15mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

NOTE: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

9.1.7 Wash down the inside of the beaker and the watchglass with reagent water. Dilute the sample digestate to 50mL with reagent water in a clean 50mL graduated cylinder. Transfer the digest to a labeled storage container, usually a 125-mL plastic vial.

NOTE: The digestate may be diluted to a volume less than the original volume if sample concentration is required to meet lower reporting limits. The pre-concentration should be limited to a factor of four(4).

9.1.8 Record the analyst's initials, the final volume of the sample digestate, and the date and time that the digestion was completed in the digestion logbook. The sample is now ready for analysis by ICP.

9.2 Total Metals: Aqueous Samples

This digestion procedure is used for the preparation of aqueous samples for total metal determination by ICP (or flame AA). This digestion procedure is not suitable for samples which will be analyzed by graphite furnace atomic absorption (GFAA) because hydrochloric acid (HCl) can cause interferences during furnace atomization.

9.2.1 Using a graduated cylinder, transfer a 50-mL aliquot (or an appropriate volume diluted to 50-mL with reagent water) of a well-mixed sample to a clean 250-mL Teflon beaker or other suitable digestion vessel.

NOTE: If there is not sufficient volume to use a 50-mL aliquot, the lab can use a smaller volume of sample and bring the final digestate volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digestate should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

9.2.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

9.2.3 Add 0.50mL of the appropriate spiking solutions to a 50-mL aliquot of reagent water designated as the laboratory control spikes(LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
6010	Liquids (aqueous)	LCS/MS	(Spex) ICP Spike I ICP Spike II

9.2.4 Add 0.50mL of the appropriate spiking solutions to two 50mL aliquots of the client sample designated as the matrix spikes samples(MS and MSD).

Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the volume of sample used
- the lot number of the acids used for the digestion
- the lot numbers of the ICP LCS spiking solutions and the ICP matrix spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE:A DIGESTION BATCH CONSISTS OF TWENTY FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. THE BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE(LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE(IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). IF THERE IS NOT SUFFICIENT SAMPLE FOR MS/MSD, THE LCS IS PREPARED IN DUPLICATE(LCSD).

9.2.5 Add 1.5mL of concentrated HNO_3 to each sample. Cover each beaker with a watch glass(a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 15mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

NOTE: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

9.2.6 Remove the beakers from the hot plate and cool the beakers to room temperature. Add another 1.5mL portion of concentrated HNO_3. Replace the watchglass and continue heating the sample on the hot plate. Again, at the proper temperature, the sample should gently reflux in the beaker-do not allow the sample to boil.

9.2.7 Continue heating the sample and adding additional 1.5mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3. If a sample requires more than 6mL of acid to digest, contact the digestion lab supervisor for guidance.

9.2.8 Evaporate the digestate (covered with the watch glass) until the volume is approximately 10mL.

9.2.9 Remove the watch glass and add a 2.5mL of concentrated HCl. Replace the watchglass on the beaker and warm the sample digestate for 15 minutes.

9.2.10 Wash down the inside of the beaker and the watchglass with reagent water. Dilute the sample digestate to 50mL with reagent water in a clean 50mL graduated cylinder. Transfer the digest to a labeled storage container, usually a 125-mL plastic vial.

NOTE: The digestate may be diluted to a volume less than the original volume if sample concentration is required to meet lower reporting limits. The pre-concentration should be limited to a factor of four(4).

9.2.11 Record the analyst's initials, the final volume of the sample digestate, and the date and time that the digestion was completed in the digestion logbook. The sample is now ready for analysis by ICP.

9.3 Total Metals: TCLP Samples

This digestion procedure is used for the preparation of TCLP leachate samples for total metal determination by ICP. This digestion procedure is not suitable for samples which will be analyzed by graphite furnace atomic absorption (GFAA) because hydrochloric acid (HCl) can cause interferences during furnace atomization. Note that the LCS is spiked with the routine analytes to allow the TCLP samples to be digested along with aqueous samples. The MS for TCLP is performed only if requested.

9.3.1 Using a graduated cylinder, transfer a 20mL aliquot (or an appropriate volume diluted to 20mL with reagent water) of a well-mixed sample to a clean 30mL Teflon vial or other suitable digestion vessel.
Larger volumes may be digested at the discretion of the lab. The volume of spike solution added should be adjusted proportionately.

9.3.2 Add 20mL of extraction fluid to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling. A blank for each type of extraction fluid must be digested and analyzed.

9.3.3 Add 0.20mL of the appropriate spiking solutions to a 20mL aliquot of extraction fluid designated as the laboratory control spike(LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 20mL aliquot of extraction fluid with 0.50mL of the appropriate spiking solutions. The routine ICP spiking solution is used for the TCLP LCS because of frequent client requests for reporting more analytes from the TCLP leachate than are currently regulated. Preparing an LCS with all of the target analytes eliminates re-digestion and provides QC for the for the requested analytes.

NOTE: If both extraction fluid I and extraction fluid II are included in the batch, use extraction fluid I for the LCS and LCSD.

Analytical Method	Matrix	QC	Spiking Solutions
6010	TCLP	MS	ICP TCLP Spike I ICP TCLP Spike II
6010	TCLP	LCS	(Spex) ICP Spike I ICP Spike II

9.3.4 If MS is requested, add 2.0mL of each ICP TCLP spiking solution (I and II) to a separate 20mL aliquot of the client sample designated as the matrix spike.

Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the volume of sample used
- the lot number of the acids used for the digestion
- the lot numbers of the ICP spiking solutions (I and II)
- the time that the digestion was started
- the SOP/method number

NOTE:A DIGESTION BATCH CONSISTS OF TWENTY FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. THE BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE(LCS), AND A MATRIX SPIKE IF REQUESTED.

9.3.5 Add 0.60mL of concentrated HNO_3 to each sample. Cover each beaker with a watch glass(a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 5mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

NOTE: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

9.3.6 Remove the beakers from the hot plate and cool the beakers to room temperature. Add another 0.60mL portion of concentrated HNO_3. Replace the watchglass and continue heating the sample on the hot plate. Again, at the proper temperature, the sample should gently reflux in the beaker-do not allow the sample to boil.

9.3.7 Continue heating the sample and adding additional 0.60mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3. If a sample requires more than 6mL of acid to digest, contact the digestion lab supervisor for guidance.

9.3.8 Evaporate the digestate (covered with the watch glass) until the volume is approximately 5mL.

9.3.9 Remove the watch glass and add a 1.0mL of concentrated HCl. Replace the watchglass on the beaker and warm the sample digestate for 15 minutes.

9.3.10 Wash down the inside of the beaker and the watchglass with reagent water. Dilute the sample digestate to 20mL with reagent water in a clean graduated cylinder. Transfer the digest to a labeled storage container, usually a 20mL plastic scintillation vial.

9.3.11 Record the analyst's initials, the final volume of the sample digestate, and the date and time that the digestion was completed in the digestion logbook. The sample is now ready for analysis by ICP.

10.0 PROCEDURES

The digestion procedure is described in Section 9.0. The analytical procedure is given in SL SOP ME 70: *Elements by ICP.*

11.0 CALCULATIONS

Calculations for the determination of metals by ICP are given in SL SOP ME70:*Elements by ICP*

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch for aqueous samples consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The analytical batch for TCLP samples consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (extraction fluid) blank, a lab control standard (LCS) spiked into the extraction fluid, and a matrix spike (MS) if requested. If both types of TCLP extraction have been used for samples in the batch, a method blank is processed for each extraction fluid and the LCS is prepared in extraction fluid I.

12.3 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE-No items in this revision.

14.0 TROUBLESHOOTING-No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 Methods 3005 and 3010: *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

15.3 Method 200.7 (Drinking Water): *Methods for the Determination of Metals in Environmental Samples*, May 1995, Supplement 1. (EPA 600/R-94/111).

15.4 Method 200.7 (NPDES): *Methods for Chemical Analysis of Water and Wastes*; USEPA Office of Research and Development, Cincinnati, Ohio, March 1982; (EPA 600/4-79-020) or latest 40CFR Part 136 citation.

Approval Signature: R. Wayne Robbins

Title: Corporate QA Manager Date: July 6, 1998

DIGESTION PROCEDURES FOR ICP TOTAL METALS IN SOILS, SEDIMENTS,WASTES, AND OILS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used to digest soil, sediment, waste, and oil samples prior to the analysis by ICP (SL SOP ME70).

2.0 SUMMARY OF METHOD

2.1 A known weight (1-2g) of the well mixed sample is transferred to a Teflon beaker or suitable digestion vessel. The sample is digested with aliquots of nitric acid and hydrogen peroxide to break down the organics present in the sample. After sample has digested, as evidenced by a clear, pale yellow digestate, HCl is added to give an approximate acid concentration of 1% HCl and 5% HNO3 and the sample digest is diluted to 100mL with reagent water.

A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits.

2.2 The SOP is based on the guidance in SW-846 Method 3050B.

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 samples are digested in strong acid solutions and contain an acid concentration of 10-20% by volume. The analyst must wear protective clothing such as a lab coat or apron. The acids used in this procedure will destroy unprotected clothing. The analyst must wear proper eye protection such as lab glasses or face shield. Acid can be splashed into the eyes from many sources. Gloves must be worn to protect hands from acid burns.

3.3 The acid digestion procedures must be performed under a properly functioning fume hood. The acid fumes from the digestion can cause mild to severe respiratory problems if breathed.

3.4 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.5 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.6 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses and the safe handling instructions for these compounds.

3.7 Care must be taken when handling the digestion beakers. Before handling a vessel that has been in use, check the temperature to make sure that it is not hot. Make sure that the digestion vessels are placed on a stable platform during and after the digestion. Vibrations from the hood or an unstable platform can cause the beakers to move and possibly to fall and splatter an analyst with a hot acid solution. Hot acids can cause severe skin burns and destroy unprotected clothing.

4.0 INTERFERENCES

Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	ROUTINE CONTAINER	PRESERVATIVE	STORAGE/ HOLD TIME
Soils/ sediments	500-mL plastic	None	<6C but not frozen
Waste/Oils	500-mL plastic or glass*	None	ambient or <6C but not frozen

*some organic wastes may destroy plastic containers

6.0 APPARATUS AND MATERIALS

6.1 Teflon or Pyrex beakers, 150-mL and 250mL

6.2 Watch glasses, to fit over beakers

6.3 Teflon vials-25mL

6.4 Hot plates-capable of maintaining a sample temperature of 95C+/-5C. The hot plates are calibrated each quarter. The temperature of 50mL water contained in a digestion vessel at the center of the hot plate is measured and the hot plate setting is marked when the temperature of the water reaches 85C. The temperature of the material in the vessel will rise to approximately 90-95C when a watch glass is placed on top. (This procedure for hot plate calibration is adopted from EPA Methods 200.7 and 200.9, which reference both aqueous and soil/solid digestions)

6.5 Volumetric flasks- 100-mL,

6.6 Graduated cylinder-100mL

6.7 Pipettes

6.8 Analytical balance

6.9 Top-loading balance

7.0 REAGENTS

Reagents must be tracked in accordance with SL SOP AN44: *Reagent Traceability*.

7.1 Reagent water-lab generated deionized water. ASTM Type I or Type II. The conductivity must be checked daily in accordance with SL SOP AN35: *Conductivity Checks for Laboratory Deionized Water*.

7.2 Nitric acid (HNO_3)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals. Each lot of acid must be assayed in the laboratory to ensure that each particular lot can be used for trace analysis.

7.3 Nitric acid solution (1:1)- Measure 500ml of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500ml of concentrated nitric acid (HNO_3) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled container suitable for storing acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed.
CAUTION: HEAT WILL BE EVOLVED AS THE NITRIC ACID MIXES WITH THE WATER. THIS SOLUTION IS WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING.

7.4 Hydrochloric acid(HCl)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals. Each lot of acid must be assayed in the laboratory to ensure that each particular lot can be used for trace analysis.

7.5 Hydrochloric acid solution (1:1)- Measure 500ml of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500ml of concentrated hydrochloric acid (HCl) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled storage container suitable for acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed.
CAUTION: HEAT WILL BE EVOLVED AS THE HYDROCHLORIC ACID MIXES WITH THE WATER. HYDROCHLORIC ACID HAS A SUFFOCATING ODOR AND MUST BE USED UNDER THE HOOD. THIS SOLUTION IS WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING. PREPARE THIS SOLUTION UNDER A HOOD.

7.6 Hydrogen peroxide, 30%-reagent grade. Check for impurities by the analysis of a method blank.

8.0 STANDARDS

The preparation of the spiking solutions 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 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Ci \otimes Vi = Cf \otimes Vf$$

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

where
Vi = volume of stock standard needed to prepare the spiking solution (mL)
Ci = concentration of stock solution (ug/mL)
Cf = concentration of spiking solution to prepare (ug/mL)
Vf = volume of spiking solution to prepare (mL)

The concentration can be expressed in whatever terms the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for Ci and Cf.

8.2 Preparation of the ICP Matrix Spiking Solutions

8.2.1 ICP Matrix Spiking Solution 1 is a solution purchased from SPEX. The catalogue number is SPIKE-1. Store this solution at room temperature. Prepare this solution every six months or sooner if needed or required.

8.2.2 Preparation of the ICP Matrix Spiking Solution 2

Add 20-mL to 30-mL of reagent water to a clean100-mL volumetric flask. Add 1-mL of concentrated nitric acid (HNO_3) and 5-mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume.

Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of std. (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100

Dilute to a final volume of 100-mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

9.0 SAMPLE PREPARATION

This digestion procedure is used for the preparation of soil, sediment, waste, and oil samples for total metal determination by ICP (or flame AA). This digestion procedure is not suitable for some analytes that will be analyzed by graphite furnace atomic absorption (GFAA) because HCl can cause interferences during furnace atomization.

9.1 Weigh 1g to 2-g (wet weight) of a homogeneous sample into a 125-mL Teflon beaker or other suitable digestion vessel. The lab may weigh a larger aliquot equal to 1.0g of sample on a dry weight basis.

NOTE: A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits; if 0.1g is digested, the final volume of the digestate must be 10mL. If the sample weight: final volume ratio is greater than 1:100, the reporting limits will be higher than those listed in the CQAP.

9.1.1 To homogenize a soil sample, the sample may be vigorously stirred in the sample container or transferred a plastic "baggie" and thoroughly mixed by kneading the container. After the sample is homogenized, return only enough sample to the original container to fill it three fourths full. This will allow the sample to be stirred and homogenized if additional aliquots of the sample are required. Place the discarded sample in a containerized waste receptacle for disposal.

9.1.2 If the sample is a solid material, break up the solid in the baggie by hitting the sample with a hammer or other suitable crushing device. Contact the immediate supervisor if the matrix is difficult to break up or is difficult to mix.

9.2 Add 1.0-mL of the appropriate spiking solution to the designated laboratory control spikes. The LCS/LCSD are prepared by weighing 1g aliquots of the "blank soil" into labeled beakers or digestion vessels.

9.3 Add 1.0mL of the appropriate spiking solution to the designated matrix spike samples. The MS/MSD are prepared by weighing 1g to 2g of the sample chosen for the matrix spike into labeled beakers or digestion vessels. The lab may weigh a larger aliquot equal to 1.0g of sample on a dry weight basis for the MS/MSD or may use a smaller weight if the final volume of the digestate is adjusted or if higher reporting limits are acceptable.

9.4 Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the weight of sample used
- the lot number of the acids used for the digestion
- the lot number of the ICP spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE: THE DIGESTION BATCH CONSISTS OF TWENTY OR FEWER FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. A DIGESTION BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE(LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE(IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). PERFORM THE LCS IN DUPLICATE IF THE MS/MSD CANNOT BE PERFORMED. THE METHOD BLANK IS PERFORMED WITH THE REAGENTS USED FOR THE DIGESTION. LAB SPIKES FOR SOIL MATRICES WILL BE PERFORMED USING 1-G ALIQUOTS OF BLANK SAND.

9.5 Add 5mL of reagent water and 5mL of concentrated HNO_3 to each beaker, mix, and cover the beaker with a watchglass.

9.6 Carefully heat the beaker until a gentle reflux is achieved-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Do not allow the samples to boil. Reflux for 10-15 minutes.

9.7 Remove the beakers from the hot plate and allow the beakers to cool to room temperature. Add 5mL of concentrated HNO_3 to each sample. Replace the watchglass and return the beakers to the hot plate. Carefully heat the beaker until a gentle reflux is achieved. Reflux the samples for 30 minutes. Do not allow the samples to boil.

9.8 Repeat the procedure in Step 9.7 with a second 5-mL portion of concentrated HNO_3 if brown fumes are given off. Repeat 9.7 until no brown fumes are given off.

9.9 Evaporate the sample digestate to approximately 10mL. Do not allow the bottom of the beaker to go dry during the evaporation. Allow the sample to cool to room temperature before continuing onto the next step.

NOTE: If the sample is still warm when the 30% H_2O_2 (hydrogen peroxide) is added in the next step, the sample may "boil over" and the entire process must be started over.

9.10 Add 2mL of reagent water to each beaker. Slowly and carefully add 3mL of 30% H_2O_2 to each beaker. It is very important to add the hydrogen peroxide slowly to prevent loss of sample due to vigorous effervescence. Return the beakers to the hot plate and heat until the effervescence subsides. Cool the beaker after the effervescence subsides.

9.11 Continue to add 30% H_2O_2 in 1-3mL aliquots to the sample digestate until the effervescence is minimal or until the general appearance of the digestate is unchanged. Warm the sample digestate after each addition of H_2O_2 on the hot plate.

NOTE: Do not add more than 10mL of hydrogen peroxide to each sample.

9.12 After the last addition of peroxide, reduce the volume of the digest to 5-10mL without boiling and without allowing the bottom of the beaker to go dry. Add 10mL of concentrated HCl to each sample digestate. Replace the watch glass and return the beakers to the hot plate and reflux the sample digestates for 10-15 minutes.

9.13 Wash down the inside of the beaker and the watchglass with reagent water. Dilute the sample digestate to 100mL with reagent water in a clean 100-mL graduated cylinder. Transfer the digestate to a labeled storage container.

9.14 Record the analyst's initials, the final volume of the sample digestate, and the date that the digestion was completed in the digestion logbook. The sample is now ready for analysis.

10.0 PROCEDURES

The digestion procedure is described in Section 9.0. The analytical procedure is given in SL SOP ME 70: *Elements by ICP.*

11.0 CALCULATIONS

Calculations for the determination of metals by GFAA are given in SL SOP ME70:*Elements by ICP*

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

ME60:04.01.98:5

Approval Signature: R. Wayne Robbins

R. Wayne Robbins

Title: Corporate QA Manager Date: 4/8/98

DIGESTION PROCEDURES FOR GRAPHITE FURNACE ATOMIC ABSORPTION TOTAL METALS AND TOTAL RECOVERABLE METALS IN LIQUID SAMPLES

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used to digest water, drinking water, wastewater, and TCLP and EP leachate samples prior to analysis by graphite furnace atomic absorption (GFAA). Two digestions procedures are included: total metals and total recoverable metals. Total metals is a more vigorous digestion and must be used for 7000-series methods and TCLP and EP leachates. Total recoverable metals may be used for 200-series methods.

2.0 SUMMARY OF METHOD

2.1 Total Metals-A known volume, usually 50mL, of an aqueous or leachate sample is transferred to a Teflon beaker. The sample is refluxed with nitric acid at approximately 90-95C. After the sample has digested, as evidenced by a clear digestate, the sample is brought up to the original volume with reagent water.

2.2 Total Recoverable Metals-A known volume, usually 50mL, of an aqueous sample is transferred to a Teflon beaker. The sample is refluxed with dilute nitric acid at approximately 95C. After the sample has evaporated to approximately 15mL, the sample is brought up to the original volume with reagent water.

2.3 Drinking water samples with a turbidity concentration of less than 1 NTU can be analyzed with no digestion if the required quantitation limits can be achieved with no sample preconcentration. The exception to this rule is silver, which requires sample digestion prior to analysis.

2.4 Samples filtered for the determination of dissolved metals do not require digestion if the sample

1) has a low COD(<20mg/L);
2) has a turbidity <1 NTU ;
3) is colorless with no significant odor ; and
4) is of one liquid phase and free of suspended particulates or precipitates after acidification

(40 CFR Part 136 Table 1B-note 4)

2.5 This SOP is based on the guidance in SW-846 Method 3020A and EPA Method 200.9.

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 samples are digested in strong acid solutions and contain an acid concentration of 10-20% by volume. The analyst must wear protective clothing. The acids used in this procedure will destroy unprotected clothing. The analyst must wear proper eye protection. Acid can be splashed into the eyes from many sources.

3.3 The acid digestion procedures must be performed under a properly functioning fume hood. The acid fumes from the digestion can cause mild to severe respiratory problems if breathed.

3.4 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.5 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.6 Each analyst should be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in this procedure. These sheets denote the type of hazard that each reagent poses and the safe handling instructions for these compounds.

3.7 Care must be taken when handling the digestion beakers. Before handling a vessel that has been in use, check the temperature to make sure that it is not hot. Make sure that the digestion vessels are placed on a stable platform during and after the digestion. Vibrations from the hood or an unstable platform can cause the beakers to move and possibly to fall and splatter an analyst with a hot acid solution. Hot acids can cause severe skin burns and destroy unprotected clothing.

4.0 INTERFERENCES

Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion. Glassware must be cleaned in accordance with SL SOP AN60: *Glassware Cleaning Procedures.*

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	ROUTINE CONTAINER	PRESERVATIVE	STORAGE/HOLD TIME
Aqueous (water)	500-mL plastic	HNO3 to pH <2	Ambient.

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.

The pH of all preserved samples must be checked and documented upon arrival in the lab. If the pH is not within the proper range, additional acid is added to the sample to bring the pH below 2.

- Place a piece of pH paper (wide range or narrow range can be used) on a watch glass or other inert surface.

- Transfer a few drops of the sample to the pH paper and note the color change. If the pH <2, record this in the log and transfer the sample to the storage area.

- If the pH is greater than 2, move the sample under a hood. Add 1:1 nitric acid to the sample in 1mL aliquots, checking the sample pH after each addition, until the pH <2. The volume of 1:1 nitric acid added to the sample should not exceed 1% of the total volume of the sample. For a 500-mL sample, the maximum volume of 1:1 nitric is 5mL. If more acid is required, contact the supervisor for further guidance. An anomaly form (SL SOP CA85) is initiated for samples that require pH adjustment after arrival in the lab.

NOTE: Samples that are not at pH<2 upon arrival in the lab may contain cyanide or sulfide or may be highly buffered. Working under a hood minimizes the hazard the may be caused by the evolution of hydrogen cyanide or hydrogen sulfide upon acidification of the sample. Be aware that acid/base neutralization reactions may be violent and evolve a good deal of heat.

6.0 APPARATUS AND MATERIALS

6.1 Digestion vessels: Teflon or Pyrex beakers, 150-mL and 250mL, or comparable digestion vessels (250-mL beakers are listed in the preparation steps but 125-mL or 50-mL digestion block digestion vessels can be substituted)

6.2 Watch glasses, "ribbed", to fit over digestion vessels

6.3 Hot plates or digestion block-capable of maintaining a sample temperature of 90-95C. The hot plates or digestion block are calibrated each quarter. The temperature of 50mL of water contained in a digestion vessel at the center of the hot plate or placed in the digestion block is measured and the setting is marked when the temperature of the water reaches 85C (+/- 3C). The temperature of the material in the vessel will rise to approximately 95C +/- 5C when a watch glass is placed on top. (This procedure for calibration is adapted from EPA Methods 200.7 and 200.9, which reference both aqueous and soil/solid digestions)

NOTE: The use of hot plates is listed in the preparation steps but a digestion block may be used if the same general procedures are employed. The hot plate or digestion block settings must be recorded in the maintenance log or other suitable log.

6.4 Teflon vials-25mL

6.5 Volumetric flasks- 100-mL

6.6 Graduated cylinder-50mL

6.7 Pipettes

6.8 Analytical balance

6.9 Top-loading balance

7.0 REAGENTS

7.1 Reagent water-lab generated deionized water. ASTM Type I or Type II. The conductivity must be checked in accordance with SL SOP AN35: *Conductivity Checks for Laboratory Deionized Water.*

7.2 Nitric acid (HNO_3)-reagent grade.

7.3 Nitric acid solution (1:1)- Measure 500mL of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500mL of concentrated nitric acid (HNO_3) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled container suitable for storing acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed. CAUTION: HEAT WILL BE EVOLVED AS THE NITRIC ACID MIXES WITH THE WATER. THIS SOLUTION IS WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING.

7.4 Hydrochloric acid (HCl)- reagent grade.

7.5 Hydrochloric acid solution (1:1)- Measure 500mL of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500mL of concentrated hydrochloric acid (HCl) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled container suitable for storing acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed. CAUTION: HEAT WILL BE EVOLVED AS THE HYDROCHLORIC ACID MIXES WITH THE WATER. THIS SOLUTION IS WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING.

8.0 STANDARDS

The preparation of the spiking solutions 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 expiration date of all spiking solutions is 180 days from date of 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 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

where
Vi = volume of stock standard (or *initial standard*) needed to prepare the spiking solution(mL)
Ci = concentration of stock solution(or *initial standard*)(ug/mL)
Cf = concentration of spiking solution to prepare(*final concentration*)(ug/mL)
Vf = volume of spiking solution to prepare(*final volume*)(mL)

The concentration can be expressed in whatever terms the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for *Ci* and *Cf*.

8.2 GFAA Spiking Solution
This spiking solution contains the elements that are routinely analyzed by GFAA.

8.2.1 Add 1.0mL of nitric acid to a 100mL volumetric flask containing about 50mL of reagent water.

8.2.2 Add *Vi* of the stock standards to the flask and dilute to a final volume of 100mL with reagent water. Mix thoroughly and transfer to a labeled storage container. Prepare this solution as needed or every six months.

The following table gives a recipe for the preparation of the GFAA matrix spiking solution:

GFAA Spiking Solution

Element	*Ci(mg/L)*	*Vi(mL)*	*Vf(mL)*	*Cf(mg/L)*
Silver (Ag)	1000	0.050	100	0.50
Arsenic(As)	1000	0.50		5.0
Selenium(Se)	1000	0.50		5.0
Lead(Pb)	1000	0.50		5.0
Thallium(Tl)	1000	0.50		5.0
Cadmium(Cd)	1000	0.050		0.50
Copper(Cu)	1000	0.50		5.0
Chromium(Cr)	1000	0.50		5.0
Antimony(Sb)	1000	0.50		5.0
Nickel(Ni)	1000	0.50		5.0

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9.0 SAMPLE PREPARATION

The turbidity of drinking water samples can be checked using the procedures in SL SOP BA80:*Turbidity*. If the turbidity of the sample is not checked, the digestion procedure must be performed. If silver must be determined in a drinking water sample, the sample must be digested.

9.1 Total Metals-Aqueous Samples and TCLP

9.1.1 Using a graduated cylinder, transfer a 50-mL aliquot (or an appropriate volume diluted to 50-mL with reagent water) of a well-mixed sample to a clean 125-mL Teflon beaker or other suitable digestion vessel.

NOTE: If there is not sufficient volume to use a 50-mL aliquot, the lab can use a smaller volume of sample and bring the final digested volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digested should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

9.1.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

9.1.3 Add 0.50mL of the graphite furnace spiking solution to a 50-mL aliquot of reagent water designated as the laboratory control spikes (LCS).

9.1.4 Add 0.50mL of the graphite furnace spiking solutions to two 50mL aliquots of the client sample designated as the matrix spikes samples(MS and MSD).

9.1.5 Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the volume of sample used
- the lot number of the acids used for the digestion
- the lot numbers of the graphite furnace LCS spiking solutions and the graphite furnace matrix spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE:A DIGESTION BATCH CONSISTS OF TWENTY FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. THE BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE(LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE(IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). IF THERE IS NOT SUFFICIENT SAMPLE FOR MS/MSD, THE LCS IS PREPARED IN DUPLICATE(LCSD).

9.1.6 Add 1.5mL of concentrated HNO_3 to each sample. Cover each beaker with a watch glass (a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 15-20mL. DO NOT ALLOW THE SAMPLE TO COMPLETELY EVAPORATE TO DRYNESS!

NOTE: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

9.1.7 Remove the beakers from the hot plate and cool the beakers to room temperature. Add another 1.5mL portion of concentrated HNO_3. Replace the watch glass and continue heating the sample on the hot plate. Again, at the proper temperature, the sample should gently reflux in the beaker-do not allow the sample to boil.

9.1.8 Continue heating the sample and adding additional 1.5-mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3. If a sample requires more than 12mL of acid to digest, contact the digestion lab supervisor for guidance.

9.1.9 Evaporate the digestate (covered with the watch glass) until the volume is approximately 5 to 10mL.

9.1.10 Wash down the inside of the beaker and the watch glass with reagent water. Dilute the sample digestate to 50mL with reagent water in a clean 50-mL graduated cylinder. The sample digestate should have an acid concentration of about 1% HNO_3.Transfer the digest to a labeled storage container, usually a 125-mL plastic vial.

9.1.12 Record the analyst's initials, the final volume of the sample digestate, and the date and time that the digestion was completed in the digestion logbook. The sample is now ready for analysis by graphite furnace.

9.2 Total Recoverable Metals-Aqueous Samples (for 200-series only)

9.2.1 Using a graduated cylinder, transfer a 50-mL aliquot (or an appropriate volume diluted to 50-mL with reagent water) of a well-mixed sample to a clean 125-mL Teflon beaker or other suitable digestion vessel.

NOTE: If there is not sufficient volume to use a 50-mL aliquot, the lab can use a smaller volume of sample and bring the final digested volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digested should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

9.2.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

9.2.3 Add 0.50mL of the graphite furnace spiking solution to a 50-mL aliquot of reagent water designated as the laboratory control spikes (LCS).

9.2.4 Add 0.50mL of the graphite furnace spiking solutions to two 50-mL aliquots of the client sample designated as the matrix spikes samples(MS and MSD).

9.2.5 Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the volume of sample used
- the lot number of the acids used for the digestion
- the lot numbers of the graphite furnace LCS spiking solutions and the graphite furnace matrix spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE:A DIGESTION BATCH CONSISTS OF TWENTY FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. THE BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE(LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE(IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). IF THERE IS NOT SUFFICIENT SAMPLE FOR MS/MSD, THE LCS IS PREPARED IN DUPLICATE(LCSD).

9.2.6 The acids added to each sample will depend of the requested analysis.

9.2.6.1 EPA 200.9
Add 1.0mL of 1:1 HNO_3 and 0.50mL of 1:1 HCl to each sample. Cover each beaker with a watch glass(a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 15mL. DO NOT ALLOW THE SAMPLE TO COMPLETELY EVAPORATE TO DRYNESS!

9.2.6.2 Other 200-series methods
Add 1.0mL of 1:1 HNO_3 to each sample. Cover each beaker with a watch glass(a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 15mL. DO NOT ALLOW THE SAMPLE TO COMPLETELY EVAPORATE TO DRYNESS!

9.2.7 Wash down the inside of the beaker and the watch glass with reagent water.

9.2.7.1 EPA 200.9: Dilute the sample digestate to 25mL with reagent water in a clean graduated cylinder. Transfer the digest to a labeled storage container, usually a 125-mL plastic vial.

9.2.7.2 Other 200-series methods: Dilute the sample digestate to 50mL with reagent water in a clean graduated cylinder. Transfer the digest to a labeled storage container, usually a 125-mL plastic vial.

9.2.8 Record the analyst's initials, the final volume of the sample digestate, and the date and time that the digestion was completed in the digestion logbook. The sample is now ready for analysis by graphite furnace.

10.0 PROCEDURES

The digestion procedure is described in Section 9.0. The analytical procedure is given in SL SOP ME 75: *Graphite Furnace AA*.

11.0 CALCULATIONS

Calculations for the determination of metals by GFAA are given in SL SOP ME75: *Graphite Furnace AA*.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 Method 3020. *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC. (Update III).

15.3 EPA Method 200.9. *Methods for the Determination of Metals in Environmental Samples*, May 1994, Supplement 1. (EPA 600/R-94/111).

Approval Signature: R. Wayne Robbins

R. Wayne Robbins

Title: Corporate QA Manager Date: July 6, 1998

DIGESTION PROCEDURES FOR GRAPHITE FURNACE ATOMIC ABSORPTION TOTAL METALS IN SOILS, SEDIMENTS, WASTES, AND OILS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used to digest soil, sediment, waste, and oil samples prior to the analysis by graphite furnace atomic absorption (GFAA).

2.0 SUMMARY OF METHOD

2.1 A known weight of the well mixed sample, approximately 1g, is transferred to a Teflon beaker. The sample is digested with aliquots of nitric acid and hydrogen peroxide to break down the organics present in the sample. After the sample has been digested, the digest is diluted to a final volume of 100mL or less with reagent water.

A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits.

2.2 This SOP is based on the guidance in SW-846 Method 3050B.

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 samples are digested in strong acid solutions and contain an acid concentration of 10-20% by volume. The analyst must wear protective clothing. The acids used in this procedure will destroy unprotected clothing. The analyst must wear proper eye protection. Acid can be splashed into the eyes from many sources.

3.3 The acid digestion procedures must be performed under a properly functioning fume hood. The acid fumes from the digestion can cause mild to severe respiratory problems if breathed.

3.4 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.5 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.6 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses and the safe handling instructions for these compounds.

3.7 Care must be taken when handling the digestion beakers. Before handling a vessel that has been in use, check the temperature to make sure that it is not hot. Make sure that the digestion vessels are placed on a stable platform during and after the digestion. Vibrations from the hood or an unstable platform can cause the beakers to move and possibly to fall and splatter an analyst with a hot acid solution. Hot acids can cause severe skin burns and destroy unprotected clothing.

4.0 INTERFERENCES

Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion. Glassware must be cleaned in accordance with SL SOP AN60: *Glassware Cleaning Procedures.*

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	ROUTINE CONTAINER	PRESERVATIVE	STORAGE/ HOLD TIME
Soils/ sediments	500-mL plastic	None	<6C but not frozen
Waste/Oils	500-mL plastic or glass*	None	ambient or <6C but not frozen

*some organic wastes may destroy plastic containers

6.0 APPARATUS AND MATERIALS

6.1 Teflon or Pyrex beakers, 150-mL and 250mL

6.2 Watch glasses, to fit over beakers

6.3 Teflon vials-25mL

6.4 Hot plates-capable of maintaining a sample temperature of 90-95C. The hot plates are calibrated each quarter. The temperature of 50mL water contained in a digestion vessel at the center of the hot plate is measured and the hot plate setting is marked when the temperature of the water reaches 85C. The temperature of the material in the vessel will rise to approximately 90-95C when a watch glass is placed on top. (This procedure for hot plate calibration is adopted from EPA Methods 200.7 and 200.9, which reference both aqueous and soil/solid digestions)

6.5 Volumetric flasks- 100-mL,

6.6 Graduated cylinder-100mL

6.7 Pipettes

6.8 Analytical balance

6.9 Top-loading balance

7.0 REAGENTS

Reagents must be tracked in accordance with SL SOP AN44:*Reagent Traceability*.

7.1 Reagent water-lab generated deionized water. ASTM Type I or Type II. The conductivity must be checked in accordance with SL SOP AN35: *Conductivity Checks for Laboratory Deionized Water.*

7.2 Nitric acid (HNO_3)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals. Each lot of acid must be assayed in the laboratory to ensure that each particular lot can be used for trace analysis.

7.3 Nitric acid solution (1:1)- Measure 500ml of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500ml of concentrated nitric acid (HNO_3) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled container suitable for storing acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed. CAUTION: HEAT WILL BE EVOLVED AS THE NITRIC ACID MIXES WITH THE WATER. THIS SOLUTION IS WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING.

7.4 Hydrogen peroxide, 30%-reagent grade. Check for impurities by the analysis of a method blank.

8.0 STANDARDS

The preparation of the spiking solutions 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 expiration date of all spiking solutions is 180 days from date of 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 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Ci \otimes Vi = Cf \otimes Vf$$

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

where
Vi = volume of stock standard (or *initial standard*) needed to prepare the spiking solution(mL)
Ci = concentration of stock solution(or *initial standard*)(ug/mL)
Cf = concentration of spiking solution to prepare(*final concentration*)(ug/mL)
Vf = volume of spiking solution to prepare(*final volume*)(mL)

The concentration can be expressed in whatever terms the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for *Ci* and *Cf*.

8.2 GFAA Spiking Solution
This spiking solution contains the elements that are routinely analyzed by GFAA.

8.2.1 Add 1.0mL of nitric acid to a 100mL volumetric flask containing about 50mL of reagent water.

8.2.2 Add *Vi* of the stock standards to the flask and dilute to a final volume if 100 with reagent water. Mix thoroughly and transfer to a labeled storage container. Prepare this solution as needed or every six months. The following table gives a recipe for the preparation of the GFAA matrix spiking solution:

Element	*Ci(mg/L)*	*Vi(mL)*	*Vf(mL)*	*Cf(mg/L)*
Silver (Ag)	1000	0.050	100	0.50
Arsenic(As)	1000	0.50		5.0
Selenium(Se)	1000	0.50		5.0
Lead(Pb)	1000	0.50		5.0
Thallium(Tl)	1000	0.50		5.0
Cadmium(Cd)	1000	0.050		0.50
Copper(Cu)	1000	0.50		5.0
Chromium(Cr)	1000	0.50		5.0
Antimony(Sb)	1000	0.50		5.0
Nickel(Ni)	1000	0.50		5.0

9.0 SAMPLE PREPARATION

9.1 Weigh 1g to 2-g (wet weight) of a homogeneous sample into a 125-mL Teflon beaker or other suitable digestion vessel. The lab may weigh a larger aliquot equal to 1.0g of sample on a dry weight basis.

NOTE: A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits; if 0.1g is digested, the final volume of the digestate must be 10mL. If the sample weight: final volume ratio is greater than 1:100, the reporting limits will be higher than those listed in the CQAP.

9.1.1 To homogenize a soil sample, the sample may be vigorously stirred in the sample container or transferred a plastic "baggie" and thoroughly mixed by kneading the container.

9.1.2 If the sample is a solid material, break up the solid in the baggie by hitting the sample with a hammer or other suitable crushing device.

Contact the immediate supervisor if the matrix is difficult to break up or is difficult to mix.

9.2 Add 1.0-mL of the graphite furnace spiking solution to the laboratory control spike (LCS). The LCS is prepared by weighing 1g of the "blank soil" into a labeled beaker or digestion vessel.

9.3 Add 1.0mL of the graphite furnace spiking solution to the designated matrix spike samples. The MS/MSD are prepared by weighing 1g to 2g of the sample chosen for the matrix spike into labeled beakers or digestion vessels. The lab may weigh a larger aliquot equal to 1.0g of sample on a dry weight basis for the MS/MSD or may use a smaller weight if the final volume of the digestate is adjusted or if higher reporting limits are acceptable.

9.4 Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the weight of sample used
- the lot number of the acids used for the digestion
- the lot number of the graphite furnace spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE: THE DIGESTION BATCH CONSISTS OF TWENTY OR FEWER FIELD SAMPLES AND The ASSOCIATED QC ITEMS. A DIGESTION BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE(LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE(IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). PERFORM THE LCS IN DUPLICATE IF THE MS/MSD CANNOT BE PERFORMED. THE METHOD BLANK IS PERFORMED WITH THE REAGENTS USED FOR THE DIGESTION. LAB SPIKES FOR SOIL MATRICES WILL BE PERFORMED USING 1-G ALIQUOTS OF BLANK SAND.

9.5 Add 5mL of reagent water and 5mL of concentrated HNO_3 or 10 ml of 1:1 HNO_3 to each beaker, mix, and cover the beaker with a watch glass.

9.6 Carefully heat the beaker until a gentle reflux is achieved-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Reflux/digest the samples for 10 to 15 minutes after the samples have attained 90-95C. **Do not allow the samples to boil.**

9.7 Remove the beakers from the hot plate and allow the beakers to cool to room temperature. Add 5mL of concentrated HNO_3 to each sample. Replace the watch glass and return the beakers to the hot plate. Carefully heat the beaker until a gentle reflux is achieved. Reflux the samples for 30 minutes at 95C. Remove the samples from the hot plate and allow to cool. Do not allow the samples to boil.

9.8 Repeat the procedure in Step 9.7 with a second 5-mL portion of concentrated HNO_3. If brown fumes are given off, Repeat Step 9.7 until no brown fumes are given off.

9.9 Evaporate the sample digestate to approximately 10mL. Do not allow the bottom of the beaker to go dry during evaporation. **Allow the sample to cool to room temperature before continuing onto the next step.**

NOTE: If the sample is still warm when the 30% H_2O_2 (hydrogen peroxide) is added in the next step, the sample may "boil over" and the entire process must be started over.

9.10 Add 2mL of reagent water to each beaker. Slowly and carefully add 1mL to 3mL of 30% H_2O_2 to each beaker. It is very important to add the hydrogen peroxide slowly to prevent loss of sample due to vigorous effervescence. Return the beakers to the hot plate and heat until the effervescence subsides. Cool the beakers after the effervescence subsides.

9.11 Continue to add 30% H_2O_2 in 1-3mL aliquots to the sample digestate until the effervescence is minimal or until the general appearance of the digestate is unchanged. Warm the sample digestate after each addition of H_2O_2 on the hot plate.

NOTE: Do not add more the 10mL of hydrogen peroxide to each sample.

9.12 After the last addition of peroxide, continue heating to a final volume of 5-10 ml. Remove the beakers from the hot plate and allow to cool.

9.13 Wash down the inside of the beaker and the watch glass with reagent water. Dilute the sample digestate to 100mL with reagent water in a clean 100-mL graduated cylinder. The sample digestate should have an acid concentration of about 10%HNO_3. Transfer the digestate to a labeled storage container.

9.14 Record the analyst's initials, the final volume of the sample digestate, and the date that the digestion was completed in the digestion logbook. The sample is now ready for analysis by graphite furnace.

10.0 PROCEDURES

The digestion procedure is described in Section 9.0. The analytical procedure is given in SL SOP ME 75: *Graphite Furnace AA*.

11.0 CALCULATIONS

Calculations for the determination of metals by GFAA are given in SL SOP ME75: *Graphite Furnace AA*.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Approval Signature: R. Wayne Robbins
Title: Corporate QA Manager Date: June 19, 1998

ELEMENTS BY ICP
(200.7 and 6010B)

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures to determine the concentration of various elements by inductively coupled plasma (ICP) atomic emission spectroscopy. This method contains the analytical procedures for the determination of metals in surface and ground water, wastewater, soil, sediment, leachate (EP or TCLP), and waste samples after digestion.

1.2 Table 1 lists the elements that may be determined by ICP and the characteristic wavelength used for each element. The reporting limit (RL) for each element, the method detection limit (MDL) for each element, and the accuracy and precision criteria for each element are listed in Section 5 of the Savannah Labs' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis by ICP, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and exited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

2.2 This method is based on EPA Method 200.7 and SW-846 Method 6010B. Note that EPA has promulgated two versions of method 200.7-one for NPDES samples and one for drinking water. The calibration sequence for drinking water by 200.7 requires a multi-point curve with a minimum of three standards and a calibration blank.

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 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.3 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions.

4.0 INTERFERENCES

4.1 Spectral interferences are caused by (1) the overlap of a spectral line from another element, (2) unresolved overlap of molecular band spectra, (3) background contribution from continuous phenomena, and (4) stray light from the line emissions of highly concentrated elements.

4.1.1 Spectral overlap may be compensated for by the use of inter-element correction factors.

4.1.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample digestate, by using a peristaltic pump, or by using the method of standards additions(MSA), or use of an internal standard

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Aqueous Samples

5.1.1 Liquid samples are collected in 250mL or 500mL 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 500mL plastic containers. The sample is iced at the time of collection and is stored in the lab at 4C (less than 6C but not frozen) 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 (less than 6C but not frozen) until the mercury analysis is completed. Hold time for mercury is 28 days.

5.4 Waste Samples

Waste samples are collected in 500mL plastic containers. The sample must be digested and analyzed within 6 months of collection.

6.0 APPARATUS AND MATERIALS

6.1 Thermo Jarrell Ash ICAP-61, TJA- Enviro 36, TJA ICAP61E-trace, or other suitable inductively coupled plasma emission spectrometer with data system

6.2 Argon gas supply and appropriate fittings

6.3 Cooling water supply

6.4 Peristaltic pump

6.5 Volumetric flasks

6.6 Pipettes

7.0 REAGENTS

Reagents are tracked in accordance with SL SOP AN44:*Reagent Traceability*.

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with SL SOP AN35.

7.2 Nitric acid (HNO_3)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

7.4 Hydrochloric acid (HCl)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Table 2 lists the various stock standards that are used to prepare the calibration and QC standards. 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 Initial Calibration Standards

8.1.1 Preparation of the Calibration Blank (ICB, CCB)

The calibration blank is reagent water that has been acidified with a mixture of HCl and HNO_3. The calibration blank is used as the initial calibration blank (ICB) and the continuing calibration blank (CCB).
The calibration blank must be prepared every six months or sooner if target elements are detected above the reporting limit.

EXAMPLE: Preparation of One Liter of the Calibration Blank

Add 500mL to 600mL of reagent water to a clean 1-L volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The Calibration Blank will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Dilute to a final volume of 1.0-L with reagent water. Store the standard at room temperature.

Larger volumes may be prepared at the discretion of the lab. The nitric acid concentration must be 1% by volume and the hydrochloric acid concentration must be 5% by volume.

8.1.2 Preparation of Calibration STANDARD 2

Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (ml)	Conc. of Cal Std (mg/L)
Silver(Ag)	1000	0.50	500	1.0
Arsenic(As)	1000	0.50		1.0
Molybdenum(Mo)	1000	0.50		1.0
Lead(Pb)	1000	0.50		1.0
Selenium(Se)	1000	5.0		10
Antimony(Sb)	1000	0.50		1.0
Thallium(Tl)	1000	5.0		10

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this standard every six months if needed or required.

8.1.3 Preparation of Calibration STANDARD 3

Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Beryllium(Be)	1000	0.50	500	1.0
Barium(Ba)	1000	5.0		10
Cadmium(Cd)	1000	0.50		1.0
Cobalt(Co)	1000	0.50		1.0
Chromium(Cr)	1000	5.0		10
Copper(Cu)	1000	5.0		10
Manganese(Mn)	1000	5.0		10
Nickel(Ni)	1000	2.5		5.0
Zinc(Zn)	1000	2.5		5.0

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this standard every six months or sooner if needed or required.

8.1.4 Preparation of Calibration STANDARD 4

Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum(Al)	10000	0.50	500	10
Iron(Fe)	10000	0.50		10
Boron(B)	1000	5.0		10
Strontium(Sr)	1000	5.0		10

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this standard every six months or sooner if needed or required.

8.1.5 Preparation of Calibration STANDARD 5

Add 200mL to 300mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Calcium(Ca)	10000	0.50	500	10
Potassium(K)	10000	1.0		20
Magnesium(Mg)	10000	0.50		10
Sodium(Na)	10000	0.50		10
Tin(Sn)	1000	5.0		10
Vanadium(V)	1000	5.0		10

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this standard every six months if needed or required.

8.1.6 Preparation of Calibration Standard SiO_2

Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volume of the stock standard given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume of Cal Std (ml)	Conc. Of Stock Std (mg/l)
Silica (SiO2)	1000	1.0	100	10

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this standard ever six months if needed or required.

8.1.7 Preparation of the calibration standards for multi-point instrument calibration.

For all drinking water samples the ICP must be calibrated with a minimum of three standards and a blank. The following standards may be used for this purpose.

With the Thermo Jarrell Ash software the Calibration Analysis and Curve-fit programs must be used to be successful with the multi-point calibration of the ICP instruments.

8.1.7.1 Preparation of the High Standard.

Add 200mL to 300mL of reagent water to a clean 1000-L volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum (Al)	10000	1.0	1000	10
Antimony (Sb)	1000	1.0		1.0
Arsenic (As)	1000	1.0		1.0
Boron (B)	1000	10		10
Barium (Ba)	1000	10		10
Beryllium (Be)	1000	1.0		1.0
Cadmium (Cd)	1000	1.0		1.0
Calcium (Ca)	10000	1.0		10
Cobalt (Co)	1000	1.0		1.0
Chromium (Cr)	1000	10		10
Copper (Cu)	1000	10		10
Iron (Fe)	10000	1.0		10
Lead (Pb)	1000	1.0		10
Magnesium (Mg)	10000	1.0		10
Manganese (Mn)	1000	10		10
Molybdenum (Mo)	1000	1.0		1.0
Nickel (Ni)	1000	5.0		5.0
Potassium (K)	10000	1.0		10
Selenium (Se)	1000	10		10
Silver (Ag)	1000	1.0		1.0
Sodium (Na)	10000	1.0		10
Strontium (Sr)	1000	10		10
Thallium (Tl)	1000	10		10
Tin (Sn)	1000	10		10
Vanadium (V)	1000	10		10
Zinc (Zn)	1000	5.0		5.0

Dilute to a final volume of 1000mL with reagent water. Store this standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.1.7.2 Preparation of the Mid-Level Standard.

Prepare as the CCV is prepared (8.3).

8.1.7.3 Preparation of the Low-Level Standard.

Prepare as the RL/PQL Check Standard (8.4).

8.2 Preparation of the Initial Calibration Verification (ICV) Solution

The ICV is analyzed after the ICP is standardized and the standardization has been checked by the analysis of the calibration standards as "unknowns"(the analysis of the calibration standards as "unknowns" must agree within +/- 5% of the true concentration). The ICV solution verifies that the instrument is measuring the target elements within specified criteria. The ICV must be prepared from stock standards that are obtained from a different source than the stock standards used to prepare the calibration standards. The second source may be from a separate vendor or from a separate lot from the same vendor.

For silica this standard may be used for the ICV and CCV standard.

8.2.1 Add approximately 400mL of reagent water to a 500mL volumetric flask. Carefully add 25mL of concentrated HCl and 5mL of concentrated HNO_3 to the volumetric flask. The solution will have an acid concentration of 5% HCl and 1% HNO_3.

8.2.2 Using a calibrated pipette, transfer 5.0mL of SPEX QC-7 and 5.0mL of SPEX QC-19 to the 500mL volumetric flask. Add 0.50mL of the 1000mg/L Tin (Sn) stock standard to the 500mL volumetric flask. The tin (Sn) stock standard must be from a separate vendor than the vendor of the stock standard used to prepare the calibration standard.

8.2.3 Dilute to volume with reagent water. Store the standard at room temperature. Prepare this standard every six months or sooner if needed or required.

8.3 Preparation of the Continuing Calibration Verification (CCV) Solutions

The CCV is analyzed after every 10 samples (with a suggested maximum of 2 hours elapsed time), to verify that the ICP is detecting the target elements within the specified criteria.

Add 200mL to 300mL of reagent water to a clean 1000mL volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
Aluminum (Al)	10000	0.50	1000	5.0
Antimony (Sb)	1000	0.50		0.50
Arsenic (As)	1000	0.50		050
Boron (B)	1000	5.0		5.0
Barium (Ba)	1000	5.0		5.0
Beryllium (Be)	1000	0.50		0.50
Cadmium (Cd)	1000	0.50		0.50
Calcium (Ca)	10000	0.50		5.0
Cobalt (Co)	1000	0.50		0.50
Chromium (Cr)	1000	5.0		5.0
Copper (Cu)	1000	5.0		5.0
Iron (Fe)	10000	0.50		5.0
Lead (Pb)	1000	0.50		0.50
Magnesium (Mg)	10000	0.50		5.0
Manganese (Mn)	1000	5.0		5.0
Molybdenum (Mo)	1000	0.50		0.50
Nickel (Ni)	1000	2.5		2.5
Potassium (K)	10000	0.50		5.0
Selenium (Se)	1000	5.0		5.0
Silver (Ag)	1000	0.50		0.50
Sodium (Na)	10000	0.50		5.0
Strontium (Sr)	1000	5.0		5.0
Thallium (Tl)	1000	5.0		5.0
Tin (Sn)	1000	5.0		5.0
Vanadium (V)	1000	5.0		5.0
Zinc (Zn)	1000	2.5		2.5

Dilute to a final volume of 1000mL with reagent water. Store this standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.4 Preparation of the RL/PQL Check Standard for ICP

The RL/PQL Check standard is analyzed at the beginning and end of each analysis sequence as a check on the sensitivity of the ICP. The elements in the RL/PQL Check standard must be detected. Note that there are separate standards for ICP and ICP-trace.

8.4.1 Preparation of RL/PQL Stock A-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Silver (Ag)	1000	0.10	100	1.0
Arsenic (As)	1000	1.0		10
Cadmium (Cd)	1000	0.050		0.50
Copper (Cu)	1000	0.25		2.5
Nickel (Ni)	1000	0.40		4.0
Lead (Pb)	1000	0.50		5.0
Selenium (Se)	1000	5.0		50
Thallium (Tl)	1000	5.0		50

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature.

8.4.2 Preparation of RL/PQL Stock B-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Aluminum (Al)	10000	0.20	100	20
Boron (B)	1000	0.50		5.0
Barium (Ba)	1000	0.10		1.0
Beryllium (Be)	1000	0.050		0.50
Calcium (Ca)	10000	0.50		50
Cobalt (Co)	1000	0.10		1.0
Chromium (Cr)	1000	0.10		1.0
Iron (Fe)	10000	0.050		5.0
Magnesium (Mg)	10000	0.50		50
Manganese (Mn)	1000	0.10		1.0
Molybdenum (Mo)	1000	0.10		1.0
Sodium (Na)	10000	0.50		50
Antimony (Sb)	1000	0.50		5.0
Strontium (Sr)	1000	0.10		1.0
Tin (Sn)	1000	0.50		5.0
Vanadium (V)	1000	0.10		1.0
Zinc (Zn)	1000	0.20		2.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature.

8.4.3 Preparation of RL/PQL Stock C-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volume of the stock standard given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume of Cal Std	Conc. Of Stock Std
Potassium(K)	10000	1.0	100	100

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.4.4 Preparation of the RL/PQL Check Solution-ICP

Add 200mL to 300mL of reagent water to a clean 500mL volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

RL/PQL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-ICP	5.0	500
Stock B-ICP	5.0	
Stock C-ICP	5.0	

Dilute to a final volume of 500***mL*** with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.5 Preparation of the RL/PQL Check Standard for ICP-Trace

In addition to the solution prepared in 8.4.4, the following solution is prepared and analyzed to verify performance on the TJA-Trace.

The RL/PQL Check standard is analyzed at the beginning and end of each analysis sequence as a check on the sensitivity of the ICP. The elements in the RL/PQL Check standard must be detected. Note that there is an additional standard for the ICP-Trace.

8.5.1 Preparation of RL/PQL Stock A-Trace

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Arsenic (As)	1000	1.0	100	10
Antimony(Sb)	1000	2.0		20
Lead (Pb)	1000	0.50		5.0
Selenium (Se)	1000	1.0		10
Thallium (Tl)	1000	1.0		10

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature.

8.5.2 Preparation of the RL/PQL Check Solution-Trace

Add 200mL to 300mL of reagent water to a clean 500mL volumetric flask. Add 5.0mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

RL/PQL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-Trace	0.50	500

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.6 Preparation of the ICP Interference Check Solutions

These solutions are analyzed at the beginning and end of each analytical, as a check on the inter-element correction factors.

8.6.1 Preparation of ICP Interference Check Solution A

Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. Of Stock(mg/L)	mL of Stock Std	Final Volume(mL)	Conc. (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.6.2 Preparation of ICP Interference Check Solution AB -*ICP*

Add 100mL to 200mL of reagent water to a clean, amber 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock(mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200
Silver (Ag)	1000	0.50		1.0
Cadmium (Cd)	1000	0.50		1.0
Nickel (Ni)	1000	0.50		1.0
Lead (Pb)	1000	0.50		1.0
Zinc (Zn)	1000	0.50		1.0
Barium (Ba)	1000	0.25		0.50
Beryllium (Be)	1000	0.25		0.50
Cobalt (Co)	1000	0.25		0.50
Chromium (Cr)	1000	0.25		0.50
Copper (Cu)	1000	0.25		0.50
Manganese (Mn)	1000	0.25		0.50
Vanadium (V)	1000	0.25		0.50
Arsenic (As)	1000	0.50		1.0
Selenium (Se)	1000	0.50		1.0
Molybdenum (Mo)	1000	0.50		1.0
Thallium (Tl)	1000	0.50		1.0
Antimony (Sb)	1000	0.50		1.0
Tin (Sn)	1000	0.50		1.0

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.6.3 Preparation of ICP Interference Check Solution AB -Trace

Add 100mL to 200mL of reagent water to a clean, amber 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock(mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200
Silver (Ag)	1000	0.50		1.0
Arsenic (As)	1000	0.050		0.10
Barium (Ba)	1000	0.25		0.50
Beryllium (Be)	1000	0.25		0.50
Cadmium (Cd)	1000	0.50		1.0
Cobalt (Co)	1000	0.25		0.50
Chromium (Cr)	1000	0.25		0.50
Copper (Cu)	1000	0.25		0.50
Manganese (Mn)	1000	0.25		0.50
Nickel (Ni)	1000	0.50		1.0
Lead (Pb)	1000	0.025		0.050
Antimony (Sb)	1000	0.30		0.60
Selenium (Se)	1000	0.025		0.050
Thallium (Tl)	1000	0.050		0.10
Vanadium (V)	1000	0.25		0.50
Zinc (Zn)	1000	0.50		1.0
Molybdenum (Mo)	1000	0.50		1.0
Tin (Sn)	1000	0.50		1.0

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.7 Preparation of the ICP Matrix Spiking Solutions

8.7.1 ICP Matrix Spiking Solution 1 is a solution purchased from SPEX. Table 5 lists the components of SPEX SPIKE 1. Store this solution at room temperature. Prepare this solution every six months or sooner if needed or required.

8.7.2 Preparation of the ICP Matrix Spiking Solution 2

Add 20mL to 30mL of reagent water to a clean100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.8 Preparation of the IDL/MDL-ICP Solution

The IDL/MDL solution is used in this procedure for two purposes:

1) To determine the Instrument Detection Limit (IDL) of each target compound on a quarterly basis; and
2) To determine the Method Detection Limit (MDL) of each target compound as defined in SL SOP CA90.

8.8.1 Preparation of IDL/MDL Stock A-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Silver (Ag)	1000	0.10	100	1.0
Arsenic (As)	1000	1.0		10
Cadmium (Cd)	1000	0.10		1.0
Copper (Cd)	1000	0.10		1.0
Nickel (Ni)	1000	0.20		2.0
Lead (Pb)	1000	0.40		4.0
Selenium (Se)	1000	1.0		10
Thallium (Tl)	1000	1.0		10

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.8.2 Preparation of IDL/MDL Stock B-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Aluminum (Al)	10000	0.10	100	10
Boron (B)	1000	0.25		2.5
Barium (Ba)	1000	0.040		0.40
Beryllium (Be)	1000	0.010		0.10
Calcium (Ca)	10000	0.050		5.0
Cobalt (Co)	1000	0.10		1.0
Chromium (Cr)	1000	0.10		1.0
Iron (Fe)	10000	0.020		2.0
Magnesium (Mg)	10000	0.050		5.0
Manganese (Mn)	1000	0.020		0.20
Molybdenum (Mo)	1000	0.20		2.0
Antimony (Sb)	1000	0.50		5.0
Strontium (Sr)	1000	0.050		0.50
Tin (Sn)	1000	0.50		5.0
Vanadium (V)	1000	0.10		1.0
Zinc (Zn)	1000	0.20		2.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.8.3 Preparation of IDL/MDL Stock C-ICP

Add 20mL to 30mL of reagent water to a clean100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Potassium (K)	10000	1.0	100	100
Sodium (Na)	10000	0.10		10

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.8.4 Preparation of the IDL/MDL Check Solution-ICP

Add 200mL to 300mL of reagent water to a clean 1000mL volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

IDL/MDL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-ICP	10	1000
Stock B-ICP	10	
Stock C-ICP	5.0	

Dilute to a final volume of 1000mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

The IDL/MDL-ICP Check Solution contains the following elements at the given concentrations:

Element	Concentration(mg/L)
Be	0.0010
Mn	0.0020
Ba	0.0040
Sr	0.0050
Ag, Cu, Co, V, Cd, Cr	0.010
Fe, Mo, Zn, Ni	0.020
B	0.025
Pb	0.040
Ca, Mg, Na, Sn, Sb	0.050
Al, As, Se, Tl	0.10
K	0.50

8.9 Preparation of the IDL/MDL-Trace Solution

The IDL/MDL solution is used in this procedure for two purposes:

1) To determine the Instrument Detection Limit (IDL) of each target compound on a quarterly basis; and
2) To determine the Method Detection Limit (MDL) of each target compound on an annual basis.

8.9.1 Preparation of IDL/MDL Stock A-Trace

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Silver (Ag)	1000	0.040	100	0.40
Arsenic (As)	1000	0.20		2.0
Barium (Ba)	1000	0.020		0.20
Beryllium (Be)	500	0.010		0.050
Cadmium (Cd)	1000	0.040		0.40
Lead (Pb)	1000	0.10		1.0
Antimony (Sb)	1000	0.20		2.0
Selenium (Se)	1000	0.20		2.0
Thallium (Tl)	1000	0.20		2.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.9.2 Preparation of IDL/MDL Stock B-Trace

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Cobalt (Co)	1000	0.030	100	0.30
Chromium (Cr)	1000	0.10		1.0
Copper (Cu)	1000	0.10		1.0
Manganese (Mn)	1000	0.020		0.20
Molybdenum (Mo)	1000	0.040		0.40
Nickel (Ni)	1000	0.10		1.0
Tin (Sn)	1000	0.20		2.0
Vanadium (V)	1000	0.060		0.60
Zinc (Zn)	1000	0.10		1.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.9.3 Preparation of IDL/MDL Stock C-ICP

Add 20mL to 30mL of reagent water to a clean100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Aluminum (Al)	10000	0.20	100	20
Calcium (Ca)	10000	0.10		10
Iron (Fe)	10000	0.10		10
Magnesium (Mg)	10000	0.10		10
Potassium (K)	10000	0.20		20
Sodium (Na)	10000	0.040		4.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.9.4 Preparation of the IDL/MDL Check Solution-Trace

Add 200mL to 300mL of reagent water to a clean 1000mL volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

IDL/MDL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-Trace	5.0	1000
Stock B-Trace	5.0	
Stock C-Trace	5.0	

Dilute to a final volume of 1000mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

The IDL/MDL-Trace Check Solution contains the following elements at the given concentrations:

Element	Concentration(mg/L)
Be	0.00025
Ba, Mn	0.0010
Co	0.0015
Ag, Cd, Mo	0.0020
V	0.0030
Cr, Cu, Ni, Pb, Zn	0.0050
As, Sb, Se, Sn, Tl	0.010
Na	0.020
Ca, Fe, Mg	0.050
Al, K	0.10

8.10 Preparation of the Linearity Check Solutions

The linearity check solutions are prepared individually according to the following equation:

$$Vs = \frac{Vlc \otimes Clc}{Cs}$$

where
Vs = volume of stock standard (mL)
Cs = concentration of stock standard (mg/L)
Vlc = volume of linearity check standard to prepare (mL)
Clc = concentration of linearity check standard to prepare (mg/L)

The linearity check solutions are prepared at the concentrations specified in Table 1. Prepare sufficient volume to perform the linearity check, maintaining the hydrochloric acid concentration at 5% by volume and the nitric acid concentration at 1% by volume.

9.0 SAMPLE PREPARATION

The sample preparation and digestion procedures are listed in the following SOPs:

MATRIX	SL SOP
Aqueous and leachate samples	ME50
Soils and Sediments	ME51
Wastes and oils	ME51

10.0 ANALYSIS PROCEDURE

10.1 Calibration/Standardization of the ICP

10.1.1 Initial Calibration/Standardization

10.1.1.1 The ICP is turned on and allowed to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If optics were turned off allow 2 hours warm up time.

10.1.1.2 Radial plasma ICP: A 1000mg/L Yttrium standard is aspirated into the plasma and the flow of the argon into the plasma is adjusted. The "red flame tongue" inside the plasma should be at the same height as the glass cylinder surrounding the torch at the proper argon flow. The argon flow will be about 0.6 L/minute. As the nebulizer ages, the flow may have to be increased or the nebulizer cleaned/replaced.

10.1.1.3 Mirror adjustment-for ICPs equipped with "D-shaped" mirror. This will entail a horizontal profile of the "D" shaped mirror using the manual profile program maximizing for intensity.

10.1.1.4 Run the "Automatic Profile" program. The "automatic profile" of the instrument should be checked twice a day to compensate for changes in air pressure, humidity, and temperature. If the environment of the instrument is such that daily changes in the instrument profile are extreme, the instrument should be "profiled" every few hours.

10.1.1.5 Analyze the calibration standards and calibrate the ICP. ***If using a multi-point calibration, use the Calibration/Analysis and Curvefit programs to calibrate the instrument.***

10.1.1.6 The highest concentration calibration standard is reanalyzed after the instrument is standardized as an "unknown". The results for the re-analysis of the highest concentration calibration standard must be within +/- 5% of the true value for each target analyte.. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

10.1.1.7 The QC Check standards (ICV) and the Calibration Blank (ICB) are analyzed as a check on the instrument calibration.

10.1.1.7.1 (EPA Method 6010) The results for the target compounds in the initial calibration verification (ICV) must be within the +/- 10 % of the true value.

10.1.1.7.2 (EPA Method 200.7) The results for the target compounds in the initial calibration verification (ICV) must be within the +/-5.0 % of the true value. When performing 200.7 work, note that this solution should be prepared fresh weekly.

10.1.1.7.3 (EPA 6010/200.7) The results for the target compounds in the initial calibration blank (ICB) must be less than the COMPQAP Table 5 Reporting Limit (RL).

10.1.1.8 The RL/PQL Check Solution is analyzed. The results for the analysis of the RL/PQL Check solution are not checked against a set of criteria but is analyzed to demonstrate that the ICP is capable of detecting the target compounds at or near the reporting limit (RL).

10.1.1.9 The ICP Interference Check Sample is analyzed.

10.1.2 Continuing Calibration

10.1.2.1 The calibration of the ICP must be verified every 10 samples by the analysis of the analysis of the QC Check Solutions (CCV) and the Calibration Blank (CCB).

10.1.2.1.1 (EPA Method 6010/200.7-DW) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/- 10 % of the true value.

10.1.2.1.2 (EPA Method 200.7-NPDES) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/-5.0 % of the true value.

10.1.2.1.3 (EPA 6010/200.7) The results for the target compounds in the continuing calibration blank (CCB) must be less than the Reporting Limit (RL).

10.1.2.2 ICP Interference Check Solution and the RL check solution are analyzed at the beginning and end of each analytical sequence.

10.2 Sample Analysis

10.2.1 The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.2.2 The samples are analyzed in a sequence as follows:

INSTRUMENT WARM-UP
ARGON FLOW ADJUSTMENT-1000mg/L Yttrium (Y)
MIRROR ADJUSTMENT
PROFILE
INITIAL CALIBRATION (STANDARDIZATION/*CALIBRATION* OF THE ICP)
REANALYSIS OF HIGH CONCENTRATION CALIBRATION STANDARD AS A SAMPLE
INITIAL CALIBRATION VERIFICATION (ICV)
INITIAL CALIBRATION BLANK (ICB)
DETECTION LIMIT CHECK SOLUTION
ICP INTERFERENCE CHECK SOLUTION A (ICSA)
ICP INTERFERENCE CHECK SOLUTION AB (ICSAB)
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB

The analytical sequence must end with the analysis of the detection limit check standard, ICSA, ICSAB, CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.2.3 Determine the concentration of the samples and QC items using the procedures of Section 11.

10.2.3.1 If the concentration of a sample is above the linear range of the ICP, the sample digestate must be diluted and reanalyzed.

10.2.3.2 The amount of sample digestate needed to prepare the desired dilution is determined form the following equation:

$$V_{digest} = \frac{Vf_{vol}}{DF}$$

where
Vf_{vol} = final volume of diluted sample (mL)
V_{digest} = volume of sample digestate used to make the dilution (mL)

10.2.3.3 The dilution factor is calculated as follows:

$$DF = \frac{Vf_{vol}}{V_{digest}}$$

where
Vf_{vol} = final volume of diluted sample extract (mL)
V_{digest} = volume of sample extract used to make the dilution (mL)

NOTE: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE
A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$DF = \frac{100mL}{1.0mL} = 100$$

Dilutions are prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

10.3 Dilution QC Check

A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

10.3.1 Select a sample digestate that contains one or more target analytes at a concentrations greater than 10X the reporting limit.

10.3.2 Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

10.3.3 Compare the results of the diluted and un-diluted aliquots of sample digestate.

10.3.4 If the results of the dilution are within ± 10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than than ± 10%, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike (see section 10.4).

If the concentration of the analyte in the sample is not at least 50 times the instrument detection limit, evaluate the post-digestion spike.

10.4 Post-digestion Spike QC Check
A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. This should be the same sample selected for dilution in 10.3, above.

10.4.1 Transfer 10mL of a digestate to a suitable vial.

10.4.2 Spike the sample with 0.10mL of ICP Matrix Spike I and 0.10mL of ICP Matrix Spike II. The theoretical concentration of the post digestion spike is the same as the LCS or MS if the volume of spiking solution is discounted.

10.4.3 Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed).

10.4.4 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \times 100$$

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)

10.4.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 75-125% recovery.

Result of Post Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<75% 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. Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples) If recoveries are not 75-125%, analyze all associated samples by single point MSA. Note – high level of target analytes may inhibit spike recovery. Consult the supervisor in events where high levels of targets appear to be interfering

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high.

10.4.6 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 response. The analyst must use good judgement when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyzed to determine the extract of the matrix interferences.

10.5 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, Vx, are taken. One aliquot is spiked with a solution of known concentration, Cs. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The concentration 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

S_1 = absorbance or concentration of the spiked aliquot
S_2 = absorbance or concentration of the un-spiked aliquot
Vs = Volume of spike solution

Example:

Sample concentration (S_2):	523 ug/L.
Spike solution concentration (C_s):	50,000 ug/L
Volume of spike solution (V_s):	0.10mL
Volume of sample aliquots (V_x):	10mL
Spiked sample concentration (S_1):	951 ug/L

C_x = [(523)*(0.10)*(50,000)]/[(951-523)*10)] = [2,615,000]/[4280] = 611 ug/L

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Aqueous and Leachate Samples

Aqueous samples are routinely reported in mg/L while the ICP 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:

$$Concentration\ (mg/L) = ug/L(from\ printout)\ x \frac{F}{V}\ x\ DF \otimes \frac{1mg}{1000ug}$$

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 CQAP 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 ICP 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:

$$Concentration(mg/kg,dw) = RL_{qap} \otimes \frac{0.0010kg}{W \otimes solids} \otimes \frac{F}{0.100L} \times DF$$

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

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 difference between the MDL and the IDL is the *digestion step*. The MDL samples are prepared and digested prior to analysis. 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.

12.4 For instrument calibration, if any fit, other than linear, is utilized for the calibration of the ICP (i.e., Curvilinear or Full Fit) the upper limit of the linear range is the concentration of the High Standard.

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.

15.4 ***Methods for the Determination of Metals in Environmental Samples***; US EPA Office of Research and Development. Washington, DC.

METHOD SUMMARY - ICP ANALYSIS

HOLD/STORAGE

Container	Minimum 250mL plastic bottle with 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 must be stored at 4C (less than 6C but not frozen) until preparation.
Hold Time	Samples may be held for up to six months from the time of collection.

SAMPLE PREPARATION

Samples should be prepared with the appropriate matrix-specific procedure.

ANALYTICAL SEQUENCE

Ignite Plasma	Follow instrument manufacturer's guidelines and allow instrument to stabilize for at least 60 minutes.
Check viewing height	If applicable to instrument aspirate 1000 ppm yttrium std. and make sure the yttrium "tongue" is set to be at the top of the outer cylinder of the torch to 1 - 2 mm above the torch.
Check horizontal (mirror) profile	Verify horizontal profile is maximized according to instrument manufacturer's recommended procedure.
Profile Instrument	Record the profile intensity and the caliper reading so that instrument drift can be monitored.
Initial Calibration	Calibrate with a blank and a high standard or a blank and three standards. Verify calibration by reanalyzing high standard.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the run. ICV solution must come from a source other than the calibration standard source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with concentrations at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run Analyze a continuing calibration blank after every CCV.
Interference Check Solutions	At the beginning and the end of an analysis run, verify the inter-element and background corrections by analyzing the interferent check solutions (ICSA & ICSAB).
Detection limit check solution	At the beginning and the end of an analysis run and verify the accuracy at the PQL by analyzing a solution at the SL PQL
Serial Dilution	For new or unusual matrices, perform serial dilution (1/5) on a representative sample.
Post Digestion Spike Recovery.	To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). The post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration in the sample is not at least 50 times the instrument detection limit.

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 std. and 1 blank	
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Highest Standard	Immediately after every calibration	Recoveries within ± 5% of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within ±10% 200.7 = within ± 5%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10% of the true value, 200.7-NPDES - within ± 5% 200.7-Drinking Water – within ±10%	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 that the PQL/CRDL	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning and end of an analysis run	Determined values must be within ± 20% of the true values. Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Lab control sample	One per batch of twenty samples or less	6010B: SL QAP 200.7: 85-115%	Redigest and reanalyze batch
Preparation blank - SW846	One per batch of twenty samples or less	|result| <PQL or result <5% of the analyte level in the sample.	Redigest and reanalyze batch
Preparation blank - 200.7	One per batch of twenty samples or less	|result| <CRDL/PQL or result <10% of the analyte level in the sample	Redigest and reanalyze batch
MS/MSD - SW846	One set per batch of twenty samples or less	SL QAP	Flag and report data
1/5 Dilution	One per batch of twenty samples or less	See section 10.3.4	
Post Digestion Spike	One per batch of twenty samples or less	See section 10.4.5	
Detection Limit Check Solution	At the beginning and end of an analysis run	Detection	Stop the analysis, fix the problem and reanalyze the affected samples.

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
				ICP/Trace	ICP/Trace	Water (mg/L)	Soil (mg/kg)
Aluminum (Al)	308.215	10	1.0/5.0	0.20/0.20	750/800	2.0	200
Antimony (Sb)	206.838	10	1.0/0.50	0.050/0.020	50/10	0.50	50
Arsenic (As)	189.042 193.696	1.0	1.0/0.50	0.10/0.010	50/25	2.0	200
Barium (Ba)	493.409	10	1.0/5.0	0.010/0.010	100/10	2.0	200
Beryllium (Be)	313.042	1.0	1.0/0.50	0.0050/0.0050	30/10	0.050	5.0
Boron (B)	249.678	10	1.0/5.0	0.050	100	1.0	100
Cadmium (Cd)	226.502 228.802	1.0	1.0/5.0	0.0050/0.0050	30/10	0.050	5.0
Calcium (Ca)	317.933 315.887	10	1.0/5.0	0.50/0.50	1000/800	5.0	500
Chromium (Cr)	267.716	10	1.0/5.0	0.010/0.010	100/25	0.20	20
Cobalt (Co)	228.616	1.0	1.0/0.50	0.010/0.010	100/25	0.50	50
Copper (Cu)	324.754	10	1.0/5.0	0.025/0.025	100/50	0.25	25
Iron (Fe)	259.940 271.441	10	1.0/5.0	0.050/0.050	800/800	1.0	100
Lead (Pb)	220.353	1.0	1.0/0.50	0.050/0.0050	100/5	0.50	50
Lithium (Li)							
Magnesium (Mg)	279.079	10	1.0/5.0	0.50/0.50	1000/1000	5.0	500
Manganese (Mn)	257.610	10	1.0/5.0	0.010/0.010	100/50	0.50	50
Molybdenum (Mo)	202.030	1.0	1.0/0.50	0.010/0.010	50/50	0.50	50

SL SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC.

TABLE 1							
Element	**Wavelength (nm)**	**Calibration Conc. (mg/L)**	**ICV/CCV Conc. (mg/L)**	**RL Std. Conc. (mg/L)**	**Linear Range Std. Conc. (mg/L)***	**MATRIX SPIKE CONC. (mg/L)**	
				ICP/Trace	ICP/Trace	**Water (mg/L)**	**Soil (mg/kg)**
Nickel (Ni)	231.604	5.0	1.0/2.5	0.040/0.040	100/10	0.50	50
Phosphorus(P)							
Potassium (K)	766.491	20	10/5.0	1.0/1.0	1000/50	5.0	500
Selenium (Se)	196.026	10	1.0/5.0	0.10/0.010	50/25	2.0	200
Silica (SiO_2)	251.611	10	1.0/5.0	0.50	100	5.0	500
Silver (Ag)	328.068	1.0	1.0/5.0	0.010/0.010	30/5.0	0.050	5.0
Sodium (Na)	588.995	10	1.0/5.0	0.50/0.50	1000/20	5.0	500
Strontium (Sr)	421.552	10	1.0/5.0	0.010	100	0.50	50
Thallium (Tl)	189.042 190.801 377.572	10	1.0/5.0	0.50/0.010	30/30	2.0	200
Tin (Sn)	189.989	10	1.0/5.0	0.050	100/50	1.0	100
Titanium (W)							
Vanadium (V)	292.402	10	1.0/5.0	0.010/0.010	50/50	0.50	50
Zinc (Zn)	213.856	5.0	1.0/2.5	0.020/0.020	100/20	0.50	50

*For guidance only-instrument sensitivity will vary.

SL SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC.

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager Date: April 29, 1998

RECEIPT, LOG NUMBER ASSIGNMENT, AND DISTRIBUTION OF FIELD SAMPLES

1.0 SCOPE AND APPLICATION

This SOP describes the routine procedures used for receiving samples into the lab and checking the integrity of the samples, assignment of a sequential laboratory identification number to the samples in a given project, and the distribution of the sample to the appropriate laboratory department.

2.0 SUMMARY OF METHOD

2.1 Upon arrival in the lab, the shipping containers are opened and inspected. The temperatures of the containers are measured and recorded and the contents of the shipping containers are checked against the chain-of-custody (COC) forms. If the integrity of the shipping containers or the sample containers has been compromised or if there is a discrepancy between the COC and samples, an anomaly report is filed out by the sample receipt custodian and the assigned project manager is notified. A sequential laboratory identification number is assigned by division in order of sample receipt from the sample receipt registry. This number is recorded on the COC and on each sample container for a given project. The samples are distributed to the appropriate laboratory department where samples are relinquished to the department and the preservation is checked by departmental analysts. The samples are then properly stored until preparation and/or analysis.

2.2 Evidence of documentation of sample collection, shipment, laboratory receipt, and custody is accomplished utilizing a chain-of-custody record (Figure 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 secured area restricted to authorized personnel.

The procedures of this SOP are in accordance with this definition of custody.

3.0 PROCEDURE

3.1 Safety

3.1.1 Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.1.2 Field samples may contain hazardous waste. Caution should be exercised when opening the coolers and handling the sample containers. A strong odor from a cooler or sample container is most likely the only indicator that the sample receipt personnel will have about the nature of the sample. The receiving area supervisor and the safety officer should be immediately alerted to such samples and action taken to ensure that the samples are handled safely.

3.1.3 Many of the samples received by the lab will contain preservatives. If contacted, these preservatives can burn

unprotected skin or destroy unprotected clothing. Sample receipt personnel must wear a lab coat or apron, eye protection, and latex gloves when handling coolers or sample bottles. Sample receipt personnel must be familiar with the color codes system and the preservatives that are used in samples. The following table includes information about the preservatives:

Sample Preservative Color Code System

Special sampling containers are designated for each analyses group in the laboratory. A preservative color-code system is utilized to alert sampling teams, sample receiving personnel, and analysts handling the containers as to which preservative was added to stabilize the sample during shipment and storage. The color-coded preservative system is as follow:

PRESERVATIVE COLOR CODE KEY

RED	CAUTION! STRONG OXIDIZER! CONTAINS NITRIC ACID. AVOID skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
GREEN	CAUTION! CONTAINS SULFURIC ACID. AVOID skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
BLUE	CAUTION! STRONG CAUSTIC! CONTAINS SODIUM HYDROXIDE. AVOID skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
TAN	Contains Zinc Acetate. AVOID skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
YELLOW	Contains Sodium Thiosulfate. Sterilized container.
LT. BLUE	CAUTION! CONTAINS HYDROCHLORIC ACID! AVOID skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.

3.2 Sample Collection, Preservation, and Handling

Samples should be checked and distributed to the appropriate department as soon as possible. Short hold time parameters (Figure 2) and samples with RUSH status should be given priority for distribution.

3.3 Apparatus and Materials

Thermometer or measuring device calibration checked against a NIST-certified thermometer in accordance with SL SOP AN55:*Laboratory Thermometer Calibration.*

3.4 Receipt of Samples

The process of receiving samples into the lab and taking custody of the samples is a dynamic process. Some procedures listed in this section may be performed concurrently, although listed in different subheadings. Coolers may arrive in the lab via common courier (Federal Express, UPS, etc.), via client delivery, via courier, or via SL field crew. It may be beneficial (but not required) to record the tracking numbers of samples received via common courier.

NOTE: The lab may be requested by some clients to use a checklist that documents the receipt of samples. Sample receipt personnel must be aware of these client-specific requests.

NOTE: Foreign soils must be handled in accordance with SL SOP AN80: *Sterilization of Foreign Soils* Each foreign soil container must have a fluorescent green sticker reading affixed to the outside that reads **"FOREIGN SOIL - STERILIZE BEFORE DISPOSAL".**

3.4.1 Inspect the general condition of the coolers. Coolers with custody seal should be checked to ensure that the seal is in place. All coolers should be inspected for signs of tampering and damage. If the custody seal has been compromised or if signs of damage or tampering are evident, the assigned project manager should be notified immediately and the situation noted in an anomaly report (Figure 3).

3.4.2 Coolers that are known to contain RUSH status samples and/or samples with short hold times (Figure 2) should be checked first. Open the coolers and inspect the general condition of the contents. It is a good practice to work on the coolers on a project-by-project basis to minimize the time that samples remain at room temperature conditions.

Upon receipt, check the coolers for:

- sample container temperature
- the presence of a chain-of-custody form.
- the presence of broken or damaged containers
- containers with no labels or labels that are not legible
- sample containers suspected of being radioactive or as required by a specific program will be screened using a Ludlum Survey Meter (Model #3) and the results recorded in the sample registry.

3.4.2.1 Determine the temperature of the samples by measuring the temperature of the "temperature bottle" (usually a 100ml plastic vial filled with water). Allow the temperature device to equilibrate (not to exceed one minute) and record the temperature on the sample registry (Figure 4). If the temperature is not <6C with no frozen samples, the assigned project manager must be notified and an anomaly report must be filled out.

NOTE: The acceptance temperature for coolers is less than 6C with no frozen samples. This criteria replaces the previous criteria of 4C +/- 2C. This criteria is used as the default criteria for the generation of an anomaly report. Some states, programs, and clients may specify other acceptance criteria

If a temperature bottle is not included in the cooler, determine the temperature of the samples by inserting a thermometer between one sample container and the bubble-pack. If the samples are not wrapped in bubble-pack, wrap the sample with bubble-pack and insert the thermometer between the bubble-pack and the container. Allow the thermometer to equilibrate for two minutes. Record the temperature on the sample registry. If the temperature is not less than 6C with no frozen samples, the assigned project manager must be notified and an anomaly report filled out. The absence of a temperature bottle and the method of determining the temperature must be recorded on the anomaly report.

NOTE: To avoid compromising the integrity of the samples, sample contents/opened containers are not to be

used for temperature checks unless written instructions are delivered to the receiving area prior to sample receipt.

3.4.2.2 If no COC has been included in the cooler or has not been sent with the project samples, the sample receipt personnel must notify the assigned project manager and initiate a chain-of-custody using the SL form (Figure 1). This deficiency must be noted in an anomaly report.

3.4.2.3 If sample containers have been broken or damaged beyond use, the sample receipt personnel must notify the assigned project manager and initiate an anomaly report.

3.4.2.4 If the sample containers are not labeled or if the sample identification cannot be determined, the sample receipt personnel must notify the assigned project manager and initiate an anomaly report.

3.4.3 Assign a laboratory identification number (log number) to the project from the sample registry and record the log number on the COC. The log number will be written or affixed to each sample in the project.

3.4.4 Compare the contents of the cooler to the COC. If there is no discrepancy between the COC and the samples, the COC is signed to indicate agreement. If there are discrepancies between the COC and samples, notify the assigned project manager and fill out an anomaly report.

3.4.5 Fill out the sample registry (Figure 4). The sample registry and the COC or COC/anomaly report should agree to the number and types of containers in a given project.

3.4.7 Write or affix the log number on each sample container in the project.

NOTE: Do not use a Sharpie pen in the receiving area. These pens contain volatiles which may contaminate samples. A "Peel-off China Marker" (Faber Castell 2663) or equivalent "grease" pen may be used.

3.5 After the samples have been checked and assigned a log number, distribute the samples to the appropriate department. The transfer of samples from the sample receipt personnel to the department is documented on the Sample Internal Custody Log (Figure 5). Interdivisional sample custody is documented on the Remote Division Sample Internal Custody Log (Figure 6).

FIGURE 1

Serial Number 92438

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 (Specify)

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	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			

ORIGINAL

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FIGURE 2 - ANALYSIS WITH EXPEDITED HOLDING TIME		
Analysis	**Holding Time**	**Matrix**
MBAS, surfactants	48 hours	Water
PO_4, Orthophosphate	48 hours	Water
NO_2 - Nitrite	48 hours	Water
NO_3 - Nitrate	48 hours	Water
BOD/CBOD (Biochemical Oxygen Demand)	48 hours	Water
Turbidity	48 hours	Water
Fecal, Total Coliform (Environmental)	6 hours	Water
Fecal, Total Coliform (Drinking Water)	30 hours	Water
Fecal, Streptococci	6 hours	Water
Sulfite	Do immediately	Water
pH	Do immediately	Water
Total Residual Chlorine	Do immediately	Water
Color	48 hours	Water
Temperature	Do immediately	Water
D.O. - Dissolved Oxygen	Do immediately	Water
Free Liquid 9095 Method (Paint Filter)	48 hours	Semi-solid
Odor	6 hours	Water
SC (Specific Conductivity)	24 hours	Water
Sulfide	7 days	Water
Hexavalent Chromium (Cr6+)	24 hours	Water
TDS/TSS/TS (Total Dissolved Solids, Total Suspended Solids, Total Solids)	7 days	Water
Ferrous Iron (Fe^{2+})	24 hours	Water
Formaldehyde	24 hours	Water
Reactivity (Releasable Sulfide/Releasable Cyanide)	As soon as possible	Water/Solid
Tedlar Bags - EPA 18	72 hours	Air
3030C Digestion (acid extractable)	72 hours	Water
Oxidation Reduction Potential (ReDox or ORP)	Do immediately	Water/Solid
Delta Spread Plate Count	6 hours	Water/Solid
Heterotrophic Plate Count	6 hours	Water/Solid
Phenol Plate Count	6 hours	Water
Presence-Absence Coliform	6 hours	Water
MNO-MUG	6 hours	Water
Unpreserved VOC (no blue dot)	7 days	Water
603/8030 (Acrolein/Acrylonitrile)	3 days	Water

FIGURE 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
☐ Custody seals broken
☐ Sample container breakage
☐ Incomplete COC
☐ Cooler temp >6°C or frozen
☐ Sample container partially filled
☐ Sample received not listed on COC
☐ 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

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FIGURE 4

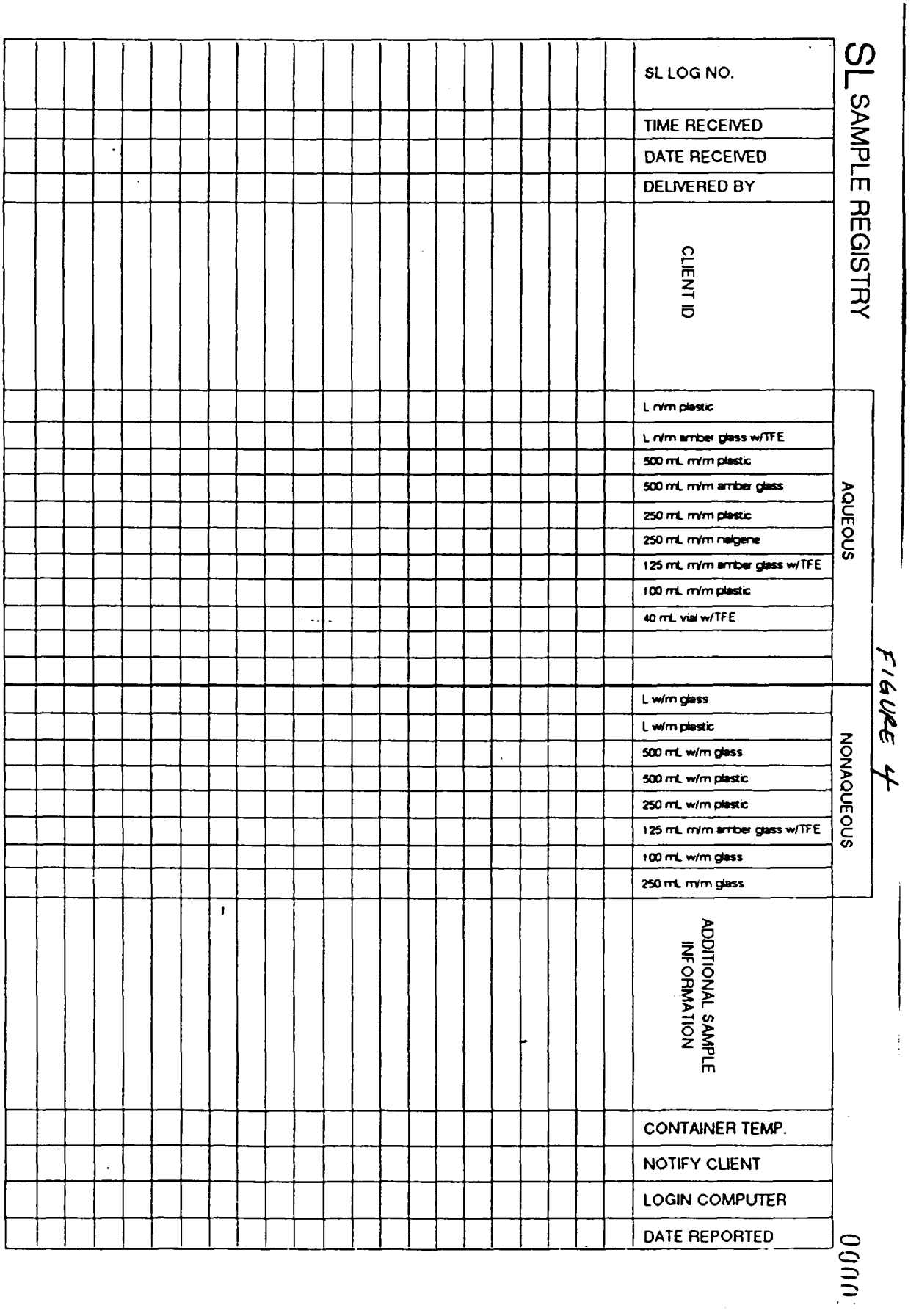
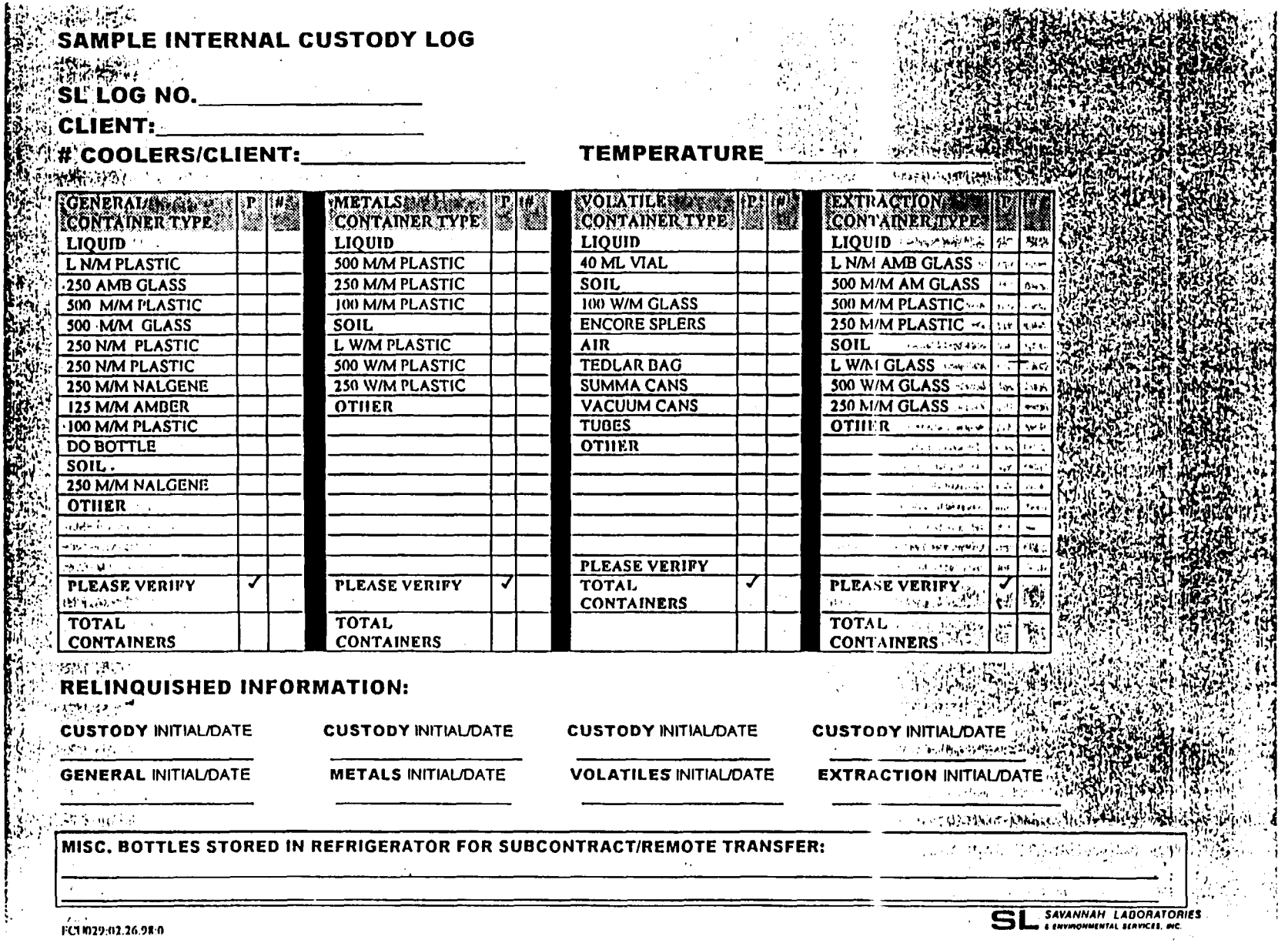

SL SAMPLE REGISTRY
SL LOG NO.
TIME RECEIVED
DATE RECEIVED
DELIVERED BY
CLIENT ID
AQUEOUS
L n/m plastic
L n/m amber glass w/TFE
500 mL n/m plastic
500 mL n/m amber glass
250 mL n/m plastic
250 mL n/m nalgene
125 mL n/m amber glass w/TFE
100 mL n/m plastic
40 mL vial w/TFE
NONAQUEOUS
L w/m glass
L w/m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

FIGURE 5



SAMPLE INTERNAL CUSTODY LOG

SL LOG NO._____

CLIENT:_____

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

GENERAL CONTAINER TYPE	P	#
LIQUID		
L N/M PLASTIC		
250 AMB GLASS		
500 M/M PLASTIC		
500 M/M GLASS		
250 N/M PLASTIC		
250 N/M PLASTIC		
250 M/M NALGENE		
125 M/M AMBER		
100 M/M PLASTIC		
DO BOTTLE		
SOIL		
250 M/M NALGENE		
OTHER		
PLEASE VERIFY	✓	
TOTAL CONTAINERS		

METALS CONTAINER TYPE	P	#
LIQUID		
500 M/M PLASTIC		
250 M/M PLASTIC		
100 M/M PLASTIC		
SOIL		
L W/M PLASTIC		
500 W/M PLASTIC		
250 W/M PLASTIC		
OTHER		
PLEASE VERIFY	✓	
TOTAL CONTAINERS		

VOLATILE CONTAINER TYPE	P	#
LIQUID		
40 ML VIAL		
SOIL		
100 W/M GLASS		
ENCORE SPLERS		
AIR		
TEDLAR BAG		
SUMMA CANS		
VACUUM CANS		
TUBES		
OTHER		
PLEASE VERIFY		
TOTAL CONTAINERS	✓	

EXTRACTION CONTAINER TYPE	P	#
LIQUID		
L N/M AMB GLASS		
500 M/M AM GLASS		
500 M/M PLASTIC		
250 M/M PLASTIC		
SOIL		
L W/M GLASS		
500 W/M GLASS		
250 M/M GLASS		
OTHER		
PLEASE VERIFY	✓	
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:

FCM029:02.26.98:0

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REMOTE DIVISION

SAMPLE INTERNAL CUSTODY LOG (Figure 6)

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 _____

SL SAVANNAH LABORATORIES ENVIRONMENTAL SERVICES, INC

Approval Signature:	R. Wayne Robbins
Title: Corporate QA Manager	Date: July 10, 1998

INTERNAL CHAIN-OF CUSTODY

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used for the distribution and tracking of samples that require a more detailed level of custody than described in SL SOP CU01:*Receipt, Log Number Assignment, and Distribution of Field Samples.*

2.0 SUMMARY OF METHOD

Upon arrival in the lab, the shipping containers are opened and inspected according to the procedures described in SL SOP CU01. A sample tracking label is affixed to each sample container and the samples are distributed to the appropriate laboratory department. The samples are relinquished to the department, and if required, the samples are checked by departmental analysts for proper preservation. The sample is then properly stored until preparation and/or analysis. Documentation is provided on the sample tracking label, sample preparation logs, the internal chain-of-custody logs, and the sample/waste disposal logs. The sample tracking labels and section internal chain-of-custody logs are maintained in department-specific notebooks.

3.0 PROCEDURE

3.1 Initiation of Internal Custody-Custody Department Personnel

3.1.1 Log samples into the lab using the procedures described in SL SOP CU01.

3.1.2 After samples are received and documented, use the LIMS to print out sample labels for each sample container. It is important that each sample have its own label. The sample log number, client ID, sample ID, and sample location items should be completed at this time.

3.1.3 Write an alpha character on the label and on the sample container to uniquely identify the container. For example, if six 1-L containers are submitted for sample MW-1, one container is designated A, another B, and so forth, with the last container designated F. See Table 1 for examples of the label and the labeling method.

3.1.4 Attach the label to the appropriate sample and distribute the samples to the departments in accordance with Section 3.5 of SL SOP CU01. The Sample Internal Custody Log (SL SOP CU01, Figure 5) is used to document the transfer of the samples from the receiving department to the analytical departments (EX, ME, VM/VG, etc.).

3.2 LIMS Log-in Status and Worksheets-Project Managers and Assistants

Samples that require a higher level of internal chain-of-custody documentation will be designated by an I status code in the LIMS. The work for these samples will be flagged on every LIMS-generated worksheet and status sheet.

3.3 Sample Chain-of Custody-Department Custody Personnel and Analysts

3.3.1 The Sample Internal Custody Log (SL SOP CU01, Figure 5) is signed by the analytical department custody personnel. This log is the initial documentation for receiving the samples into the department.

2.1.1 If required, check the sample preservation and document as described in the appropriate analytical SOP. Transfer the samples to the designated storage area.

3.3.3 When the sample is removed from the storage area, write the date and time that the sample was removed and initial the sample label. For example, if the sample was removed on June 6, 1998 at 10:00am by WR, the label will be documented :

Log Number: S880001
Client: Alpha Industries
Sample ID: MW-1 A

LOCATION: EX

OUT: **6/6/98 10a.m. WR** IN:

OUT: IN:

OUT: IN:

OUT: IN:

EMPTY:

DISPOSAL:

2.1.2 If the sample is returned to the storage area, write the date and time that the sample was returned on the sample label and initial the label. For example, if the sample was returned on June 6, 1998, at 11:10am by WR, the label will be documented :

Log Number: S880001
Client: Alpha Industries
Sample ID: MW-1 A

LOCATION: EX

OUT: 6/6/98 10:00a.m. WR IN: **6/6/98 11:10am WR**

OUT: IN:

OUT: IN:

OUT: IN:

EMPTY:

DISPOSAL:

3.3.5 After the entire contents of the sample container has been used, write the date on the sample label beside EMPTY and initial the label.

Log Number: S880001
Client: Alpha Industries
Sample ID: MW-1 B

LOCATION: EX

OUT: 6/6/98 10:00a.m. WR IN:

OUT: IN:

OUT: IN:

OUT: IN:

EMPTY: **6/6/98 WR**

DISPOSAL:

If the container is used for multiple analyses, the label may have several OUT/IN dates, times, and initials before the sample is depleted. Transfer the label to the department notebook designated for the maintaining the labels and dispose of the sample container.

3.3.6 If the sample container has not been used for analysis or if sample remains in the container, write the date that the sample was removed from the storage area for disposal on the sample label and initial the label. Transfer the sample to the designated area for disposal.

Log Number: S880001
Client: Alpha Industries
Sample ID: MW-1 F

LOCATION: EX

OUT: IN:

OUT: IN:

OUT: IN:

OUT: IN:

EMPTY:

DISPOSAL: **06/21/98 WR**

2.2 Extracts and Leachates

Metals and general lab digestates are not tracked since refrigeration is not required. The digestion matrix is stable and not prone to evaporation or degradation if analyzed within the recommended hold times.

3.4.1 If internal custody is required for TCLP or other leachates that will not be analyzed on the same day as the leaching procedure or filtration step, the procedures described for field samples are employed. A sample label is affixed to the storage container to track the sample from the leaching procedure through the final preparation (extraction or digestion).

NOTE: If a leachate must be retained for multiple analyses over several days, a sample label must be used.

3.4.2 Internal custody of extracts will be tracked on the form given in Table 2. Each time an extract is removed from the designated storage refrigerator, the date and time of removal must be recorded and initialed. If the extract is returned to the storage refrigerator, the date and time of return is recorded and initialed.

The internal chain-of custody forms are retained in the prep or analytical sections (VG/VM, EX, GE, etc.) in designated notebooks. The forms are filed by batch number and sample identification.

Table 1-Example of Labels for a Sample With Multiple Containers of the Same Type

Log Number: S880001 Client: Alpha Industries Sample ID: MW-1 A LOCATION: EX OUT: IN: OUT: IN: OUT: IN: EMPTY: DISPOSAL:	Log Number: S880001 Client: Alpha Industries Sample ID: MW-1 B LOCATION: EX OUT: IN: OUT: IN: OUT: IN: EMPTY: DISPOSAL:
Log Number: S880001 Client: Alpha Industries Sample ID: MW-1 C LOCATION: EX OUT: IN: OUT: IN: OUT: IN: EMPTY: DISPOSAL:	Log Number: S880001 Client: Alpha Industries Sample ID: MW-1 D LOCATION: EX OUT: IN: OUT: IN: OUT: IN: EMPTY: DISPOSAL:
Log Number: S880001 Client: Alpha Industries Sample ID: MW-1 E LOCATION: EX OUT: IN: OUT: IN: OUT: IN: EMPTY: DISPOSAL:	Log Number: S880001 Client: Alpha Industries Sample ID: MW-1 F LOCATION: EX OUT: IN: OUT: IN: OUT: IN: EMPTY: DISPOSAL:

TABLE 2

INTERNAL CHAIN OF CUSTODY

Batch No _____ Prep Method _____

Job No 1818 Extract Box ID / Location _____

Analysis Required _____ *THIS SECTION MUST BE FILLED OUT COMPLETELY

SDG # Sample ID / Sample Description	Client Code	PM	OUT Tm/Dt	IN Tm/Dt	OUT Tm/Dt	IN Tm/Dt	OUT Tm/Dt	IN Tm/Dt	OUT Tm/Dt	IN Tm/Dt	Dispose Date
CAP174 1. SB81817*1/C-49835-SB102-01APR98-1340	ES-WP	LW									
Lab Blank											
Lab Spike #1											
Lab Spike #2											

INTERNAL CHAIN OF CUSTODY

Batch No _____ Prep Method _____

Job No 1818 _____ Extract Box ID / Location _____

Analysis Required _____ *THIS SECTION MUST BE FILLED OUT COMPLETELY

SDG # Sample ID / Sample Desription	Client Code	PM	OUT Tm/Dt	IN Tm/Dt	OUT Tm/Dt	IN Tm/Dt	OUT Tm/Dt	IN Tm/Dt	OUT Tm/Dt	IN Tm/Dt	Dispose Date
CAP174 1. S881817*1/C-49835-SB102-01APR98-1340	ES-WP	LW									
Lab Blank											
Lab Spike #1											
Lab Spike #2											

Approval Signature: R. Wayne Robbins

Title: Corporate QA Manager Date: June 24, 1998

PREPARATION OF SAMPLING KITS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Savannah Laboratories (SL) to prepare and ship sampling kits for field use. A sampling kit contains the coolers, pre-cleaned containers, preservatives, and custody records necessary to complete a sampling event and return the samples to SL under conditions necessary to preserve the integrity of the samples. The "container tables" in appendix A list the containers and preservative for liquids and for soils and solids. The routine containers and the preservatives for each parameter are also listed in Section 6 of the SL Quality Assurance Plans.

2.0 SUMMARY OF METHOD

Requests for sampling kits ("bottle orders") are routinely made by project managers or their assistants. The request is processed via the SL Laboratory Information Management System (LIMS) and the generated sample container request form is routed to the sample container shipping department. The appropriate containers are gathered and the proper preservatives are added to the containers. A color coded, labeled sticker (a "dot") is affixed to the container to indicate the type of preservation. The containers are packed securely into coolers along with a temperature bottle, a copy of the bottle order, bags to hold ice, and chain-of-custody (COC) forms, and custody seals, if requested. The sampling kits may be picked up directly from the lab or shipped via common courier.

3.0 PROCEDURES

3.1 The project manager or the project manager's assistant enters sample container requests into the LIMS. The request form is routed to the shipping department for preparation of the sampling kits.

The form is inspected unusual requests. The project manager or assistant is notified immediately if a sampling kit request cannot be completed.

3.2 Preparation of Sampling Kits

3.2.1 The shipping department personnel gather and inspect the appropriate containers. Some questions to ask:

- are proper labels attached to the containers?
- do the containers require special preparation or custody seals?
- are any containers broken or are obvious defects present?

Notify the project manager or assistant immediately if the container request cannot be completed.

3.2.2 Add the proper preservatives to the appropriate containers. Wear gloves, a lab coat or apron, and eye protection when adding the preservatives via the "dot code" listed in a field at the top of the form and defined at the bottom of the form. The "container tables" 2 list the chemical preservatives required for the various analyses.

These reagents must be prepared and tracked according to the guidance in SL SOP AN41:*Standard Material Traceability*.

3.2.3 The reagent water and acids used to prepare trip blanks must be verified periodically to ensure that the water and acid are free from volatiles. A mechanism must be developed by each SL division lab to document that the trip blanks (water and acid) and water sent for field and equipment blanks is free from volatiles' contamination.

3.2.4 Coolers must be packed securely to minimize or prevent breakage during shipment.

- Wrap all glass containers in bubble wrap or bubble baggies prior to packing. Plastic containers may be placed in the coolers without bubble wrap but still must be securely positioned to minimize movement during shipment.

- Containers with preservatives must be secured with "closure strips" over the cap to prevent the cap from loosening and spilling the preservative.

- The following items are placed into the cooler for shipment with the containers:
  1) Chain-of-custody form(s)
  2) Custody seals (if requested)
  3) Copy of bottle order
  4) Baggies to hold ice for shipment back to the lab (each cooler)
  5) Temperature blank container (each cooler)

3.2.5 If the containers will be used to collect "foreign soils" (see SL SOP AN80: *Sterilization of Foreign Soil Samples*), Form PPQ-550, the laboratory's Department of Agriculture soil permit, and sampling crew instructions must be shipped with the containers. Green florescent stickers reading "FOREIGN SOIL-STERILIZE BEFORE DISPOSAL" must be placed on each container used to collect foreign soils prior to shipping to the client

3.2.6 After packing, the coolers are closed, sealed with tape, and placed in a shipping box.

ROUTINE 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

Routine Containers are the pre-cleaned containers that are normally used for sampling and analysis. The containers have been tested and certified by the lab to ensure that the parameters tested are below the levels published in the current SL CQAP.

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 form 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 4C (control limits of less than 6C with no frozen samples).

The hold time is the maximum time the sample can be held prior to preparation or analysis. Some parameters have separate hold times for preparation and analysis (generally organic semivolatile procedures).

The minimum volume 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.

NOTE: There are analyses where the routine reporting limit 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. This is a non-routine request and requires consultation with the laboratory director or lab manager prior to sample analysis because the amount of surrogate and 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.

APPENDIX A
ANALYSES/CONTAINERS FOR LIQUIDS -General Lab

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 immediately	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 immediately	50mL
Temperature	120-ml P	none	analyze immediately	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 Lab

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 immediately	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
Organic Carbon (TOC)	125-ml amb G	1mL 1:1 sulfuric acid	28 days	10mL
Chlorine, total residual	250-ml amb G	none	analyze immediately	25mL

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

ANALYSES/CONTAINERS FOR LIQUIDS-General Lab

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
Oxygen, dissolved (Winkler)	BOD Bottle G	HACH DO reagent powder pillows	analyze immediately	full BOD bottle
Oxygen, dissolved (electrode)	BOD Bottle G	none	analyze immediately	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-Metals Lab

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

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

ANALYSES/CONTAINERS FOR LIQUIDS-VOC Lab-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 (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 Lab-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

(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-Semivolatile 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
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/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.

ANALYSES/CONTAINERS FOR LIQUIDS-Semivolatile 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
Dioxins and Furans (8280)	2 x 1-L amb G	none (2)	30days/45 days(3)	1-L
2,3,7,8-TCDD (613/8280)	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.

ANALYSES/CONTAINERS FOR LIQUIDS-Extraction Lab

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 LIQUIDS-Liquid Chromatography

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME	MINIMUM VOLUME REQUIRED
PAHs (610/8310)	2 x 1-L amb G	none (2)	7days/40 days (3)	1-L
Carbonyl Compounds (formaldehyde and acetaldehyde) (8315)	2 x 125 ml amb G	none	3 days/3 days(3)	100mL

(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.

ANALYSES/CONTAINERS FOR LIQUIDS-Microbiological Parameters

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% sodum thiosulfate	30 hours	100mL
Coliform, fecal and total	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodum thiosulfate	6 hours	100mL
Fecal steptococci	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodum thiosulfate	6 hours	100mL
Bacteria plate count	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodum 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

CONTAINERS FOR LIQUIDS-Radiological Parameters (send to Tampa)

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

CONTAINERS FOR LIQUIDS AND SOILS-Nitrocellulose (send to Tallahassee General Lab)

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.

CONTAINERS FOR LIQUIDS-Liquid Chromatography(Send to Tallahassee)

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	sulfuric acdi to pH<2	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 highdensity ,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
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	momocloroacetic 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(Send to Tallahassee)

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
Ethylenthiouria (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
Resourcinol (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(Send to Tallahassee)

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
Ethylenthiouria (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

(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 SOILS AND SOLIDS -General Lab

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	Hold Time	MINIMUM WEIGHT REQUIRED
Ammonia	250-mL P	none	28 days	10g
Ammonia (if distillation required)	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, 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 immediately	10g
Kjeldahl (TKN) and organic nitrogen	250-mL P	none	28 days	10g
Nitrate (autoanalyzer)	250-mL P	none	2 days	10g
Nitrate (IC)	250-mL P	none	2 days	10g
Nitrite (colorimetric)	250-mL P	none	2 days	10g
Nitrite (IC)	250-mL P	none	2 days	10g
Organic Carbon	250-mL P	none	2 days	10g
Orthophosphate	250-mL P	none	2 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.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-General Lab

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-Metals Lab

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

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

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

PARAMETER	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME (3)	MINIMUM WEIGHT REQUIRED
Purgeable Halocarbons by GC (601, 8010, 8021)	2 x 5-g Encore plus 125-mL G amber	5mL 5% sodium bisulfate solution or frozen in water (2)	14 days	1 5-g Encore plus 125-mL bulk
Purgeable Aromatic Hydrocarbons by GC (602,8020,8021)	2 x 5-g Encore plus 125-mL G amber	5mL 5% sodium bisulfate solution or frozen in water (2)	14 days	1 5-g Encore plus 125-mL bulk
Halocarbons and Aromatics (601/602, 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	1 5-g Encore plus 125-mL bulk
Volatiles by GC/MS (624/8260)	2 x 5-g Encore plus 125-mL G amber	5mL 5% sodium bisulfate solution or frozen in water (2)	14 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	1 5-g Encore plus 125-mL bulk

(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. In addition, a methanol extract is prepared from the bulk sample within 48 hours of collection for high concentration samples

(3) The hold time for VOC is 48 hours if the samples are not preserved with sodium sulfate or frozen in water within 48 hours of collection

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-Semivolatile 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
Benzidines (8270C)	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
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/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.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-Semivolatile 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	30g
Dioxins and Furans (8280)	250-mL or 500-mL amb G	none	30days/45days	30g

(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.

ANALYSES/CONTAINERS FOR SOILS AND SOLIDS-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.

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.

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
SPLP	500-mL G	none	14 days(2)	100g
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 immediately	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

(2) The hold time is the maximum time until the leaching procedure is performed; after leaching, the routine liquid hold times apply.

CHANGE -IN-PROGRESS ATTACHMENT

SOP Document No: EX30:02.19.98:1

SOP Description: Continuous Liquid-Liquid Extraction

Approval Signature: R. Wayne Robbins

Title: Corporate QA Manager Date: Aug 27, 1998

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

This CIPA is made in response to the Army Corps of Engineers audit of the Tampa Division and the Navy audit of the Savannah Division. Please note the change: addition of surrogates and matrix spikes is required prior to the pH change.

9.1 Sample Evaluation

9.1.1 Remove the samples to be extracted from the storage refrigerator. Allow the samples to equilibrate to room temperature while the appropriate glassware is gathered and prepared for the extraction. Fill out as much of the extraction log as possible prior to initiating the extraction.

9.1.2 Inspect the samples. Determine if the samples have multiple layers such as sediment or an oil layer. Consult with the supervisor or technical manager if the sample matrix is unusual or is difficult to categorize or handle.

9.1.3 Mark the level of the sample on the outside of the container. After the sample has been added to the extractor body, fill the empty container with water to the mark. Pour the water into a graduated cylinder and determine the volume. Record the volume in the extraction log to the nearest 5mL..

NOTE: If a reduced volume of sample must be extracted in order to provide sufficient sample volume to meet state or other program QC requirements, the sample is thoroughly mixed and an aliquot is measured with a graduated cylinder and poured into the extractor body. The following volumes are utilized for TCLP samples for BNA and pesticides:

TCLP PARAMETER	Volume of Sample Extracted(mL)	Final Volume (mL)
BNA by GC/MS (8270)	200	1.0
Pesticides by GC (8080)	20	10

9.2 Glassware Preparation
Process glassware and samples (Sections 9.2 through 9.4) in batches not greater than 20 field samples and QC in order to minimize the evaporation of methylene chloride into the lab.

9.2.1 Prepare the continuous liquid-liquid extraction apparatus by rinsing the extractor body, receiving flask, and condenser with acetone. Pay particular attention to the solvent return line for the presence of water drops or solids.

9.2.2 Set the extractor body on the holder. Add methylene chloride (50-100ml) to the extractor body in order to fill the drain tube and ensure that a continuous flow of solvent from the extractor body to the receiving flask is maintained.

NOTE: Inspect the solvent layer when the sample is added. Large quantities of sediment may plug the tube and extraction will not occur. The newest extractor bodies have been re-designed to reduce the amount of solvent necessary to ensure a continuous flow of solvent. Use the amount of solvent-50mL or 100mL- necessary to allow the extractor to function properly.

9.2.3 In a fume hood, add 100mL to 175mL of methylene chloride to the receiving flask and add a few boiling stones to the flask. Add the same volume to each flask in the batch. Leave the receiving flask with the methylene chloride under the hood until ready to attach it to the extractor or cover tightly with aluminum foil.

9.3 Attach the receiving flask to the extractor body and secure the heating mantle around the flask.

9.4 As soon as possible after the methylene chloride is added to the extractor, gently pour the entire sample into the extractor body, trying not to let the sample leak into the sidearm. The extractor body will be approximately ½ to 2/3 full and the methylene chloride in the solvent return should be about 3/4 of the way to the receiving flask.

NOTE: The lab has the option of using an aliquot of sample if the final volume of the extract is adjusted to compensate for the reduced volume of sample. In some cases, the volume of sample must be reduced in order to have sufficient sample to extract matrix spikes as required by a state or other program quality assurance plan or regulation. In this case, the volume of surrogate and matrix spiking solution added to the sample should also be reduced proportionately .

9.5 Add 1.0L aliquots of reagent water to each of two separate extractors to serve as the method blank and lab control sample (LCS).

EXTRACTION BATCH: An extraction bath consists of up to twenty (20) field samples and the following QC items: method blank, LCS, MS, and MSD. If insufficient sample is available to perform the MS and MSD, the LCS is extracted in duplicate(LCSD) and the extraction log is stamped: INSUFFICIENT SAMPLE AVAILABLE TO PERFORM MS/MSD.

9.6 Addition of Surrogates and Matrix Spikes
The surrogate and matrix spiking solutions are set up so that 1.0ml of the solution will be added . Be sure to use the correct solutions for the extraction of concern. The addition of the surrogates and matrix spikes should be witnessed by another analyst to ensure that the proper volume of the spiking solution is added to each sample.

9.6.1 Add 1.0ml of the surrogate spiking solution to each sample and QC item.

NOTE: If the volume of sample extracted must be reduced to provide sufficient sample to meet state or other program requirements, the volume of surrogate spiking solution must be reduced proportionately.

9.6.2 Add 1.0ml of the matrix spiking solution to each LCS, MS, and MSD.

NOTE: If the volume of sample extracted must be reduced to provide sufficient sample to meet state or other program requirements, the volume of matrix spiking solution must be reduced proportionately.

9.6.3 Adjust the pH of the samples to the range specified in Table 1. The pH should be adjusted by adding small aliquots of 10N sulfuric acid or 10N sodium hydroxide directly to the extraction body. Stir the sample with a glass rod or Teflon rod. Check the pH after each addition with wide range paper. Do not dip the paper into the sample. Use a glass rod or pipette to remove a small aliquot of sample and touch the liquid to the pH paper.

NOTE: Sulfuric acid will cause the pH of the sample to decrease; sodium hydroxide will cause the pH of the sample to increase.

9.7 Slowly add reagent water to the extractor body until the level of the liquid causes the methylene chloride to just flow over to the receiving flask.

9.8 Place the condenser securely on the extractor body. Make sure that water is flowing through the condenser and turn the heating mantle on. Observe the extraction for the first hour or so to ensure that the solvent is being boiled, condensed, and returned to the receiving flask. Extract the samples for the time listed in Table 1. The batch is labeled with the start time and the scheduled stop time. I f a single pH extraction is performed, go to 9.11; if a two-pH extraction must be performed, continue at 9.9.

9.9 If a dual pH extraction is required, turn the heating mantle "off" to stop the extraction at the appropriate time and allow the extraction vessel to cool. Adjust the pH of the sample by adding small aliquots of 10N sulfuric acid or 10N sodium hydroxide. Stir the sample with a glass rod or pipette and check the pH after each addition . Do not dip the pH paper into the sample but use a glass rod or pipette to remove a small aliquot of the sample and touch the liquid to the pH paper.

NOTE: Sulfuric acid will cause the pH of the sample to decrease; sodium hydroxide will cause the pH of the sample to increase.

9.10 Turn the heating mantle "on" and extract the sample at the second pH for the time listed in Table 1. The batch is labeled with the start time and the scheduled stop time

9.11 After the samples have extracted for the required time, turn the heating mantle "off" and allow the extraction vessel to cool to room temperature. It is important to allow the receiving flask to cool before it is removed from the extractor.

9.12 Remove the receiving flask from the extractor body and cover it with a Teflon stopper. The extract is stored under near a hood until the filtration and concentration step is performed.

9.13 Working under a hood, place a piece of filter paper into a glass funnel and add a small amount of purified sodium sulfate. Filter the extract through the glass funnel and collect the extract directly into a labeled Zymark tube. Discard the filter paper and sodium sulfate into a waste container which is kept under the hood until removed from the laboratory. The extract is now ready for concentration using the Zymark instrument(SL SOP EX50) and should be kept under a hood tightly covered with aluminum foil until the concentration step. The extract may also be concentrated using the Kuderna-Danish extract evaporation(SL SOP EX51).

NOTE: Perform the extract filtration and drying steps in batches to minimize the loss of solvent into the lab. Cover the remaining containers tightly with aluminum foil and leave under or near a hood.

After the extracts have been dried and filtered, dispose of the filter paper and sodium sulfate by putting them in a chlorinated solvent waste container that is kept under the hood.

NOTE: If the volume of sample extracted has been reduced to provide sufficient sample to meet state or other program QC requirements, the final volume of the extract must be reduced proportionately.

9.14 Rinse the ground-glass joint of the condenser with acetone and wrap in aluminum foil before placing the condenser in the rack. Collect the acetone rinsate in a separate container for disposal.

9.15 After the receiving flask has been removed from the extractor body, the methylene chloride remaining in the extractor body must be properly disposed. The extractor should be handled with minimum agitation to minimize the amount of solvent allowed to evaporate into the lab and stored in the chlorinated solvent waste container. Place the receiving flask side-arm over the chlorinated waste container. Add tap water to the extractor body to displace the methylene chloride into the waste container

The liquid remaining in the extractor body should be poured carefully down the sink and flushed continuously with water.

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
Title: Corporate QA Manager Date: Oct 28, 1997

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.***

6.1 Gas chromatograph- Hewlett-Packard (HP) 5890 or equivalent with compatible autosampler, splitless injector, and direct capillary interface. **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 extracts. The carbon traps should be changed a minimum of every three months.**

Approval Signature: R. Wayne Robbins

R. Wayne Robbins

Title: Corporate QA Manager Date: 02/20/98

CONTINUOUS LIQUID-LIQUID EXTRACTION

1.0 SCOPE AND APPLICATION

This SOP describes the general procedures for the extraction of semivolatile organic compounds (SVOC) from aqueous samples using continuous liquid-liquid extraction. This procedure is applicable to a wide range of aqueous matrices including monitoring wells, industrial wastewater, saline waters, and TCLP or EP leachates.

2.0 SUMMARY OF METHOD

2.1 A known volume of sample is adjusted to a specific pH if required, transferred to a continuous liquid-liquid extractor, and extracted using the solvent and conditions specified in Table 1. The extract is concentrated to an appropriate final volume using either the Zymark nitrogen blow-down concentrator (SL-SOP EX50) or the Kuderna Danish concentration procedure (SL-SOP EX51).

2.2 This SOP is based on the guidance in SW-846 Method 3520C and the extraction sections of the EPA 600-series methods.

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 you or others in potentially hazardous situations.

3.2 This SOP contains procedures that are designed to reduce the exposure of lab personnel to solvent vapors and to minimize the amount of solvent introduced into the lab air. All solvent transfer steps should be performed quickly and under a hood, if possible. Use the minimum amount of solvent to get the job done. Do not allow open containers of solvent or extracts to evaporate into the lab.

3.3 A lab coat or apron should be worn to protect clothing and skin from acids, bases, and solvents. Gloves must be worn to protect hands and eye protection (goggles or safety glasses) must be worn at all times in the lab. An analyst must not work alone in an isolated area.

3.4 A solvent such as methylene chloride can cause a burning sensation if it contacts the skin. The rapid evaporation of the solvent causes a rapid heat loss in the skin similar to frost bite. If solvent contracts the skin, wash the exposed skin in cold water to reduce the solvent evaporation.

3.5 The analyst should become familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in the extraction lab.

4.0 INTERFERENCES

4.1 Improperly cleaned glassware can contribute interferences to the sample extract. Material that is left inside the extractor after cleaning may show up in the next extraction. The glassware must be properly cleaned according to SL SOP AN60. After cleaning, inspect the glassware for the presence of water, especially in the small tubing of the extractor. This water can block the solvent return tube and prevent efficient extraction of the sample. A thorough acetone rinsing will help to eliminate or minimize this problem.

4.2 Sample with large amounts of sediments or particulate may clog the solvent return line. A layer of glass wool, placed in the bottom of the extractor, may be helpful when sediments or particulate are present.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Aqueous samples are routinely collected in 1-L amber glass containers equipped with Teflon-lined caps. No preservative is required and an orange dot is affixed to the container to indicate this. The sample is stored at 4°C± 2°C in the lab. The sample must be extracted within seven (7) days of collection and the extract must be analyzed within forty (40) days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Continuous liquid-liquid extractor with compatible condenser, extractor body, and receiving flask.

6.2 Heating mantle and adjustable support. The heating mantle must be connected to a rheostat to control the temperature.

6.3 Rheostat or variable transformer

6.4 Teflon stoppers to fit the receiving flask

6.5 Filter paper-grade 414, 18.5cm diameter (VWR 28320-143)

6.6 Glass funnels

6.7 Pyrex glass wool - purified by Soxhlet extraction with methylene chloride.

6.8 Disposable volumetric pipettes - 1.0-mL and 10-mL

6.9 Boiling stones-Soxhlet extracted with methylene chloride

6.10 Wide range pH paper

7.0 REAGENTS

7.1 Reagent water - lab generated deionized water

7.2 Methylene chloride - residue grade or better

7.3 Acetone - residue grade or better

7.4 Sodium hydroxide (NaOH) - reagent grade

7.5 Sodium hydroxide solution (10N): Dissolve 400g of NaOH pellets into about 500mL of reagent water contained in a 2-L beaker on a magnetic stirrer. Add the NaOH in small portions, with constant stirring, to minimize the time it takes to dissolve the pellets. A good deal of heat will be generated as the NaOH dissolves. After all 400g has been added, carefully dilute to 1000mL with reagent water. Mix the solution thoroughly and transfer to a storage container. Do not store sodium hydroxide solution in volumetric glassware or in containers with ground glass joints.

7.6 Sulfuric acid (H_2SO_4) - concentrated reagent grade

7.7 Sulfuric acid solution (1:1 v/v): Slowly and carefully add 550mL of concentrated H_2SO_4 to 500mL of reagent water contained in a 2-L beaker on a magnetic stirrer. Add the acid in small portions with constant stirring to reduce the heat evolved when the acid and water are combined. Cool and transfer the solution to a labeled storage container.

7.9 Sodium sulfate-anhydrous, granular, purified by heating for 4 hours at 400C in a shallow tray. Store in glass containers

8.0 STANDARDS

The preparation of the surrogate and matrix spiking solutions are described in SL SOP EX70: *Preparation of Semivolatile Surrogates and Matrix Spiking Solutions.* Table 2 is a summary of the spiking solutions used for the various analytical extraction methods. All preparation steps must be tracked in accordance with SL SOP AN41:*Standard Material Traceability.*

The tables included in SOP EX70 describe "recipes" that can be used to prepare the spiking solutions. The lab can prepare larger or smaller volumes if desired. The recipe used by the lab may be prepared as a "controlled posting" to prompt the analyst in the preparation of the spiking solutions. The controlled posting can be posted in the lab or placed in the extraction lab tracking log.

9.0 SAMPLE PREPARATION

The samples and QC items must be clearly and unambiguously labeled during each step of the extraction and analysis process. Labeling tape should be used to identify the glassware associated with each sample. Solvents will erase grease pens and "Sharpies". Use of different colored labeling tape will aid the analyst in keeping up with the extracts during each step of the extraction and concentration.

9.1 Sample Evaluation

9.1.1 Remove the samples to be extracted from the storage refrigerator. Allow the samples to equilibrate to room temperature while the appropriate glassware is gathered and prepared for the extraction. Fill out as much of the extraction log as possible prior to initiating the extraction.

9.1.2 Inspect the samples. Determine if the samples have multiple layers such as sediment or an oil layer. Consult with the supervisor or technical manager if the sample matrix is unusual or is difficult to categorize or handle.

9.1.3 Mark the level of the sample on the outside of the container. After the sample has been added to the extractor body, fill the empty container with water to the mark. Pour the water into a graduated cylinder and determine the volume. Record the volume in the extraction log to the nearest 5mL..

NOTE: If a reduced volume of sample must be extracted in order to provide sufficient sample volume to meet state or other program QC requirements, the sample is thoroughly mixed and an aliquot is measured with a graduated cylinder and poured into the extractor body. The following volumes are utilized for TCLP samples for BNA and pesticides:

TCLP PARAMETER	Volume of Sample Extracted(mL)	Final Volume (mL)
BNA by GC/MS (8270)	200	1.0
Pesticides by GC (8080)	20	10

9.1.4 Adjust the pH of the samples to the range specified in Table 1. The pH should be adjusted by adding small aliquots of 10N sulfuric acid or 10N sodium hydroxide directly to the sample container. Shake the sample container to mix the sample with the acid or base. Check the pH after each addition with wide range paper. Do not dip the paper into the sample. Use a glass rod or pipette to remove a small aliquot of sample and touch the liquid to the pH paper.

NOTE: Sulfuric acid will cause the pH of the sample to decrease; sodium hydroxide will cause the pH of the sample to increase.

9.2 Glassware Preparation

Process glassware and samples (Sections 9.2 through 9.4) in batches not greater than 20 field samples and QC in order to minimize the evaporation of methylene chloride into the lab.

9.2.1 Prepare the continuous liquid-liquid extraction apparatus by rinsing the extractor body, receiving flask, and condenser with acetone. Pay particular attention to the solvent return line for the presence of water drops or solids.

9.2.2 Set the extractor body on the holder. Add methylene chloride (50-100ml) to the extractor body in order to fill the drain tube and ensure that a continuous flow of solvent from the extractor body to the receiving flask is maintained.

NOTE: Inspect the solvent layer when the sample is added. Large quantities of sediment may plug the tube and extraction will not occur. The newest extractor bodies have been re-designed to reduce the amount of solvent necessary to ensure a continuous flow of solvent. Use the amount of solvent-50mL or 100mL- necessary to allow the extractor to function properly.

9.2.3 In a fume hood, add 100mL to 175mL of methylene chloride to the receiving flask and add a few boiling stones to the flask. Add the same volume to each flask in the batch. Leave the receiving flask with the methylene chloride under the hood until ready to attach it to the extractor or cover tightly with aluminum foil.

9.3 Attach the receiving flask to the extractor body and secure the heating mantle around the flask.

9.4 As soon as possible after the methylene chloride is added to the extractor, gently pour the entire sample into the extractor body, trying not to let the sample leak into the sidearm. The extractor body will be approximately ½ to 2/3 full and the methylene chloride in the solvent return should be about 3/4 of the way to the receiving flask.

NOTE: The lab has the option of using an aliquot of sample if the final volume of the extract is adjusted to compensate for the reduced volume of sample. In some cases, the volume of sample must be reduced in order to have sufficient sample to extract matrix spikes as required by a state or other program quality assurance plan or regulation. In this case, the volume of surrogate and matrix spiking solution added to the sample should also be reduced proportionately .

9.5 Add 1.0L aliquots of reagent water to each of two separate extractors to serve as the method blank and lab control sample (LCS). The reagent water should be adjusted to the pH conditions listed in Table1.

EXTRACTION BATCH: An extraction bath consists of up to twenty (20) field samples and the following QC items: method blank, LCS, MS, and MSD. If insufficient sample is available to perform the MS and MSD, the LCS is extracted in duplicate(LCSD) and the extraction log is stamped: INSUFFICIENT SAMPLE AVAILABLE TO PERFORM MS/MSD.

9.6 Addition of Surrogates and Matrix Spikes
The surrogate and matrix spiking solutions are set up so that 1.0ml of the solution will be added . Be sure to use the correct solutions for the extraction of concern. The addition of the surrogates and matrix spikes should be witnessed by another analyst to ensure that the proper volume of the spiking solution is added to each sample.

9.6.1 Add 1.0ml of the surrogate spiking solution to each sample and QC item.

NOTE: If the volume of sample extracted must be reduced to provide sufficient sample to meet state or other program requirements, the volume of surrogate spiking solution must be reduced proportionately.

9.6.2 Add 1.0ml of the matrix spiking solution to each LCS, MS, and MSD.

NOTE: If the volume of sample extracted must be reduced to provide sufficient sample to meet state or other program requirements, the volume of matrix spiking solution must be reduced proportionately.

9.7 Slowly add reagent water to the extractor body until the level of the liquid causes the methylene chloride to just flow over to the receiving flask.

9.8 Place the condenser securely on the extractor body. Make sure that water is flowing through the condenser and turn the heating mantle on. Observe the extraction for the first hour or so to ensure that the solvent is being boiled, condensed, and returned to the receiving flask. Extract the samples for the time listed in Table 1. The batch is labeled with the start time and the scheduled stop time. I f a single pH extraction is performed, go to 9.11; if a two-pH extraction must be performed, continue at 9.9.

9.9 If a dual pH extraction is required, turn the heating mantle "off" to stop the extraction at the appropriate time and allow the extraction vessel to cool. Adjust the pH of the sample by adding small aliquots of 10N sulfuric acid or 10N sodium hydroxide. Stir the sample with a glass rod or pipette and check the pH after each addition . Do not dip the pH paper into the sample but use a glass rod or pipette to remove a small aliquot of the sample and touch the liquid to the pH paper.

NOTE: Sulfuric acid will cause the pH of the sample to decrease; sodium hydroxide will cause the pH of the sample to increase.

9.10 Turn the heating mantle "on" and extract the sample at the second pH for the time listed in Table 1. The batch is labeled with the start time and the scheduled stop time

9.11 After the samples have extracted for the required time, turn the heating mantle "off" and allow the extraction vessel to cool to room temperature. It is important to allow the receiving flask to cool before it is removed from the extractor.

9.12 Remove the receiving flask from the extractor body and cover it with a Teflon stopper. The extract is stored under near a hood until the filtration and concentration step is performed.

9.13 Working under a hood, place a piece of filter paper into a glass funnel and add a small amount of purified sodium sulfate. Filter the extract through the glass funnel and collect the extract directly into a labeled Zymark tube. Discard the filter paper and sodium sulfate into a waste container which is kept under the hood until removed from the laboratory. The extract is now ready for concentration using the Zymark instrument(SL SOP EX50) and should be kept under a hood tightly covered with aluminum foil until the concentration step. The extract may also be concentrated using the Kuderna-Danish extract evaporation(SL SOP EX51).

NOTE: Perform the extract filtration and drying steps in batches to minimize the loss of solvent into the lab. Cover the remaining containers tightly with aluminum foil and leave under or near a hood.

After the extractshave been dried and filtered, dispose of the filter paper and sodium sulfate by putting them in a chlorinated solvent waste container that is kept under the hood.

NOTE: If the volume of sample extracted has been reduced to provide sufficient sample to meet state or other program QC requirements, the final volume of the extract must be reduced proportionately.

9.14 Rinse the ground-glass joint of the condenser with acetone and wrap in aluminum foil before placing the condenser in the rack. Collect the acetone rinsate in a separate container for disposal.

9.15 After the receiving flask has been removed from the extractor body, the methylene chloride remaining in the extractor body must be properly disposed. The extractor should be handled with minimum agitation to minimize the amount of solvent allowed to evaporate into the lab and stored in the chlorinated solvent waste container. Place the receiving flask side-arm over the chlorinated waste container. Add tap water to the extractor body to displace the methylene chloride into the waste container

The liquid remaining in the extractor body should be poured carefully down the sink and flushed continuously with water.

10.0 PROCEDURES

No items in this revision.

11.0 DATA ANALYSIS/CALCULATIONS

No items in this revision.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Table 1 -Surrogate and Matrix Spiking Solutions

METHODS	TYPES OF COMPOUNDS	SURROGATE SPIKING SOLUTION	MATRIX SPIKING SOLUTION
604	Phenols by GC-FID	SG ACID	604
8040/8041	Phenols by GC-FID	SG ACID	8040
606,8060,8061	Phthalate esters by GC-FID	SG BASE	606/8060
607,8070	Nitroaromatics by GC-FID	8141	target compounds
608	Chlorinated pesticides and PCBs	PEST	608
8080,8081	Chlorinated pesticides and PCBs	PEST	8080
8080,8081	PCBs	PEST	PCB
8080,8081	Chlorinated pesticides-TCLP	PEST	TCLP-PEST
609,8090	Nitrosamines by GC-FID	SG BASE	target compounds
610	PAH by GC-FID	SG BASE	610
8100	PAH by GC-FID	SG BASE	8100
611,8110	Halogenated ethers by GC/EC	PEST	target compunds
612,8120	Chlorinated hydrocarbons	PEST	target compunds
614,614.1,622, 622.1	Phosphorous and nitrogen containing pesticides	8141	target compunds
8140,8141	Phosphorous and nitrogen containing pesticides	8141	8141
617	Chlorinated pesticides	PEST	target compunds
619/620	Phosphorous and nitrogen containing pesticides	8141	target compunds
627	Chlorinated pesticides	PEST	target compunds
625	BNA	625/8270	625
8270-TCL	BNA	625/8270	8270
8270-K001	BNA	625/8270	K001(8270)
8270-TCLP, AP9	BNA	625/8270	TCLP-8270
8015-EXT	Petroleum products	SG BASE	Diesel-8015-mod Mineral spirits-8015-mod

Table 2 -Extraction Conditions

METHODS	EXTRACTION CONDITIONS	EXTRACTION TIME(hr.)	SOLVENT	FINAL SOLVENT	FINAL VOLUME(mL)
604,8041	Single pH < 2	>12	MeCl2	MeCl2	1.0
606,8061	Single pH as received or at pH<2	>12	Mecl2	MeCl2	1.0
607,8070	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
608,8080,8081	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
609,8090	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
610,8100	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
611,8110	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
612,8120	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
614,614.1,622, 622.1,8141	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
617	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
619	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
627	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
8270-TCL,K001	Single pH <2	>12	MeCl2	MeCl2	1.0
625, 8270-TCLP	Dual pH at pH<2 followed by pH>12	18/18	MeCl2	MeCl2	1.0
8270-AP9	Dual pH at pH<2 followed by pH>12	>12/>12	MeCl2	MeCl2	1.0
8015-EXT	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0

SL SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC

EX40:02.19.98:1

Approval Signature:	R. Wayne Robbins
Title: Corporate QA Manager	Date: 02/20/98

ULTRASONIC EXTRACTION

1.0 SCOPE AND APPLICATION

This SOP describes the process for extracting semivolatile organic compounds (SVOC) from solid matrices by ultrasonic (sonication) extraction. This procedure is applicable to soils, sediments, sludges, and wastes.

2.0 SUMMARY OF METHOD

2.1 A known weight of a sample (approximately 30g wet weight) is combined with anhydrous, purified sodium sulfate to form a free flowing, sandy mixture. A solvent, usually 1:1 acetone/methylene chloride, is added to the dried sample and the sample is extracted using an ultrasonic disrupter for three (3) minutes. The solvent is decanted and the extraction is repeated two more times. The extract is filtered and concentrated to an appropriate final volume.
Table 1 defines the extraction conditions.

NOTE: The lab has the option of extracting a sample aliquot equal to 30g on a dry weight basis.

2.2 This procedure is based on SW-846 Method 3550B.

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 This SOP contains procedures that are designed to reduce the exposure of lab personnel to solvent vapors and to minimize the amount of solvent introduced into the lab air. All solvent transfer steps should be performed quickly and under a hood, if possible. Use the minimum amount of solvent to get the job done. Do not allow open containers of solvent or extracts to evaporate into the lab.

3.3 A lab coat or apron should be worn to protect clothing and skin from acids, bases, and solvents. Gloves must be worn to protect hands and eye protection (safety glasses, goggles) must be worn at all times in the lab. An analyst should not work alone in an isolated area.

3.4 A solvent such as methylene chloride can cause a burning sensation when it contacts the skin. The rapid evaporation of the solvent causes a rapid heat loss in the skin which is similar to frost bite. If this occurs, was the exposed skin in cold water to reduce the solvent evaporation.

3.5 The analyst should become familiar with the Material Safety Data Sheets (MSDS) for each reagent used in the extraction lab.

4.0 INTERFERENCES

Improperly cleaned glassware or equipment can contribute interferences to the sample extract. A thorough acetone rinsing will help to eliminate or minimize this problem.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Soils, sediments, sludges, and wastes are collected in glass containers equipped with Teflon-lined caps. The normal container is 500-mL glass. Larger or smaller containers may be supplied. The sample is not preserved and should have an orange dot affixed to indicate this. The samples are stored at 4C ± 2C in the lab. The hold time for SVOC in solid 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 Ultrasonic disrupter: Tekmar Model or equivalent with ¾ inch horn-type titanium-tipped sonication probe. The sonicator should be capable of operating in the pulse mode at full power.

6.2 Sonabox - the sonicator should be placed in the sonabox to reduce noise. The sonabox must be placed under a fume hood.

6.3 Pre-cleaned 500ml extraction bottles (P/N SAV-145). Discard the bottles after use.

6.4 Large glass funnels

6.5 Filter paper-18.5cm

6.6 Pyrex glass wool - purchased purified or extracted with methylene chloride for a minimum of four (4) hours.

6.7 Stainless steel spatulas

6.8 Small cups or beakers for optional pH measurement.

6.9 wide range pH paper

6.10 Disposable volumetric pipets

6.11 Top-loading balance - capable of weighing to 0.1g. The balance calibration must be verified according to SL SOP AN10 each time the balance is used.

7.0 REAGENTS

7.1 Sodium sulfate, powdered and granular, anhydrous - purified by heating at 400C for four (hours) in a shallow tray.

7.2 Methylene chloride - residue grade or better

7.3 Acetone - residue grade or better

7.4 1:1 acetone/methylene chloride: Mix equal volumes of acetone and methylene chloride in an appropriate glass container.

8.0 STANDARDS

The preparation of the surrogate and matrix spiking solutions are described in SL SOP EX70: *Preparation of Semivolatile Surrogates and Matrix Spiking Solutions.* Table 2 is a summary of the spiking solutions used for the various analytical extraction methods. All preparation steps must be tracked in accordance with SL SOP AN41:*Standard Material Traceability.*

The tables included in SOP EX70 describe "recipes" that can be used to prepare the spiking solutions. The lab can prepare larger or smaller volumes if desired. The recipe used by the lab may be prepared as a "controlled posting" to prompt the analyst in the preparation of the spiking solutions. The controlled posting can be posted in the lab or placed in the extraction lab tracking log.

9.0 SAMPLE PREPARATION

9.1 Extraction Glassware Set-up

9.1.1 Remove samples to be extracted from the storage refrigerator and allow the samples to come to room temperature while the extraction glassware is being prepared.

9.1.2 Collect the appropriate glassware and rinse with acetone prior to use. Use the minimum amount of solvent to rinse the glassware.

9.1.3 Fit a filter pad into a large funnel and rinse the filter paper/funnel with acetone.

9.2 Sample Extraction

Record as much information on the extraction log as possible before beginning the extraction to minimize the number of entries required during the extraction. The extraction steps from weighing to the sonication should be carried out quickly to minimize the loss of SVOC through evaporation.

9.2.1 Open the sample container and inspect the sample. Remove any materials such as rocks, sticks, leaves, etc. Using a stainless steel spatula , thoroughly homogenize the sample, mixing in any water that may be present on top of the sediment or soil. Use a separate spatula for each sample to prevent cross-contamination of the samples.

NOTE: If it is difficult to homogenize the sample or if the sample matrix is difficult to characterize, contact the supervisor before preceding with the extraction. A careful inspection of the sample at this point can save time and effort later on in the analysis.

9.2.2 Weigh 30.0 - 30.5g of the homogenized sample into a pre-cleaned, labeled 500-mL extraction bottle. Record the weight for all samples in this batch.

NOTE: The lab has the option of weighing an aliquot of sample equal to 30g on a dry weight basis.

Weight 30g of powdered, purified sodium sulfate into each of two separate extraction vessels to serve as the method blank and lab control spike (LCS).

Weigh two additional 30g portions of a sample in the batch selected as the matrix spike (MS) and matrix spike duplicate(MSD). The MS/MSD may be specified by the client or may be chosen from the samples in the batch.

NOTE: Each extraction batch of twenty or fewer samples will have the following QC items: method blank,

LCS, LCSD, MS, and MSD. If there is not enough sample to perform the MS/MSD, the extraction log must be stamped "INSUFFIECIENT SAMPLE WAS AVAILABLE TO PERFROM THE MS/MSD" and the LCS extracted in duplicate(LCSD).

9.2.3 Add approximately 60g of powdered, purified sodium sulfate to each sample and MS/MSD and stir with a glass rod or stainless steel spatula to form a sandy, free-flowing mixture. The sodium sulfate combines with the water in the sample to "dry" the sample (remove the water). Additional sodium sulfate may be required if the sample is very wet.

NOTE: The next steps should be performed immediately after the sodium sulfate is added to minimize the loss of SVOC due to evaporation

9.2.4 Add 1.0mL of the appropriate surrogate spiking solution (Table 1) to the method blank, LCS, MS, MSD and each sample in the batch.

9.2.5 Add 1.0ml of the appropriate matrix spiking solution (Table 1) to the LCS, MS, and MSD. Place the extraction bottle under the hood near the sonicator

9.2.6 Working under a hood, immediately add 1:1 acetone/methylene chloride to the cover the sample approximately one inch above the solids. Stir the sample to break up any lumps that may have formed.

9.2.7 Check the "tune" of the sonicator using the procedure tested in SL SOP AN15: Tekmar Sonic Dismembrator Tuning Procedure.

9.2.8 Place the tip of the sonicator horn in the center of the beaker about ½ inch below the surface of the solvent but above the solid portion. More solvent may be added to bring the level to approximately one inch above the solid layer.

9.2.9 Sonicate for three (3) minutes with output control knob set at 10, mode switch to pulse, and percent duty cycle set at 50% If the sonication is properly performed, the solids and solvent will vigorously mix each time the sonicator pulses.

9.2.10 Decant the extract through the filter funnel and collect the extract in a beaker or directly in a labeled, "elongated" Zymark tube. The elongated tubes hold approximately 225mL of solvent; therefore, it is desireable to limit the volume of the three extracts to 225mL. If the sample is very wet and requires a large amount of sodium sulfate and a larger volume of solvent, the extracts are collected in separate Zyamrk tubes and combined prior to analysis to analysis.

9.2.11 Repeat steps 9.2.6 through 9.2.10 two more times with fresh portions of methylene chloride and collect the extract in the Zymark tube. Cover the Zymark tube with aluminum foil and leave it under the hood until the concentration step.

NOTE: Clean the sonicator horn between samples by rinsing with acetone. "Sonicate" an extraction bottle of acetone for 3 minutes with output control at 5 to further clean the horn (sodium sulfate may be added to the beaker to prevent cracking).

9.2.13 Dispose of extraction bottle and sample residue by placing in a chlorinated solvent waste container which is kept under a hood until removed from the laboratory.

10.0 ANALYTICAL PROCEDURE

No items in this revision.

11.0 CALCULATIONS

No items in this revision.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Table 1 -Surrogate and Matrix Spiking Solutions

METHODS	TYPES OF COMPOUNDS	SURROGATE SPIKING SOLUTION	MATRIX SPIKING SOLUTION
8040/8041	Phenols by GC-FID	SG ACID	8040
8060,8061	Phthalate esters by GC-FID	SG BASE	606/8060
8081, 8082	Chlorinated pesticides and PCBs	PEST	8080
8082	PCBs	PEST	PCB
8081	Chlorinated pesticides-TCLP	PEST	TCLP-PEST
8100	PAH by GC-FID	SG BASE	8100
8140,8141	Phosphorous and nitrogen containing pesticides	8141	8141
8270-TCL	BNA	625/8270	8270
8270-K001	BNA	625/8270	K001(8270)
8015-EXT	Petroleum products	SG BASE	Diesel-8015-mod Mineral spirits-8015-mod

Table 2 -Extraction Conditions

METHODS	TYPES OF COMPOUNDS	EXTRACTION CONDITIONS	SOLVENT	FINAL SOLVENT	FINAL VOLUME(mL)
8040,8041	Phenols by GC-FID	As received	1:1 Ace/MeCl2	MeCl2	1.0
8060,8061	Phthalate esters by GC-FID	As received	1:1 Ace/MeCl2	MeCl2	1.0
8081, 8082	Chlorinated pesticides and PCBs	As received	1:1 Ace/Hexane	Hexane	10mL
8100	PAH by GC-FID	As received	1:1 Ace/MeCl2	MeCl2	1.0mL
8140,8141	Phosphorous and nitrogen containing pesticides	As received	1:1 Ace/Hexane	Hexane	10mL
8270-TCL,K001	BNA	As received	1:1 Ace/MeCl2	MeCl2	1.0mL
8015-EXT	Petroleum products	As received	1:1 Ace/MeCl2	MeCl2	1.0mL

SL SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC.

Approval Signature: R. Wayne Robbins

Title: Corporate QA Manager Date: 11/25/97

EXTRACTION OF CHLORINATED HERBICIDES IN WATER, SOILS, AND WASTES

1.0 SCOPE AND APPLICATION

This SOP describes the process for extracting chlorinated phenoxy herbicides from aqueous samples, soils and sediments, and solid matrices.

2.0 SUMMARY OF METHOD

2.1 A known volume of aqueous sample, nominally 500mL, is transferred to a Teflon or glass separatory funnel. The sample is made basic (pH>12) by adding sodium hydroxide and allowed to hydrolyze for at least one hour. The hydrolysis step is necessary to convert herbicides in the acid or ester form to the corresponding water-soluble salt. The sample is extracted with diethyl ether to remove non-target compounds (the herbicide salts remain in the water). The sample is acidified to convert the herbicide salts to the acid form and extracted with diethyl ether. The diethyl ether extract is concentrated and the herbicides are converted to methyl esters by diazomethane esterification. The methyl esters are solubilized in hexane and analyzed by GC/electron capture (SL SOP SG65).

2.2 A known weight of a sample (approximately 30g wet weight) is acidified with HCl and combined with acidified sodium sulfate to form a free flowing, sandy mixture. 1:1 acetone/methylene chloride is added to the dried sample and the sample is extracted using an ultrasonic disrupter for three (3) minutes. The solvent is decanted, collected and the extraction is repeated two more times. The extract is concentrated and hydrolyzed with KOH using a Kuderna-Danish apparatus. The extract is transferred to a Teflon or glass separatory funnel, adjusted to pH>12 if necessary, and extracted with methylene chloride to remove non-target compounds (the herbicide salts remain in the water). The extract is treated in the same manner as aqueous sample extracts from this point forward.

NOTE: The lab has the option of extracting a sample aliquot equal to 30g on a dry weight basis. It is the responsibility of the lab to demonstrate that this extra weight of sample does not affect the extraction efficiency.

2.3 A known weight of waste, nominally 1g, is diluted to 10mL with diethyl ether. The extract is transferred to a separatory funnel containing 500mL of reagent water that has been adjusted to a pH >12 with sodium hydroxide. The sample is allowed to hydrolyze for one hour under basic conditions and is treated in the same manner as an aqueous sample from this point forward.

2.4 This procedure is based on SW-846 Method 8151A.

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 This SOP contains procedures that are designed to reduce the exposure of lab personnel to solvent vapors and to minimize the amount of solvent introduced into the lab air. All solvent transfer steps should be performed quickly and under a hood, if possible. Use the minimum amount of solvent to get the job done. Do not allow open containers of solvent or extracts to evaporate into the lab.

3.3 A lab coat or apron should be worn to protect clothing and skin from acids, bases, and solvents. Gloves must be worn to protect hands and eye protection (safety glasses, goggles) must be worn at all times in the lab. An analyst should not work alone in an isolated area.

3.4 Diethyl ether is a flammable solvent that can cause drowsiness. The extraction analyst using diethyl ether must not work alone in an isolated area of the lab. A solvent such as diethyl ether can cause a burning sensation when it contacts the skin. The rapid evaporation of the solvent causes a rapid heat loss in the skin which is similar to frost bite. If this occurs, rinse the exposed skin in cold water to reduce the solvent evaporation.

3.5 The lab should keep the minimum supply of diethyl ether. Diethyl ether will from explosive peroxides if stored in the lab for long periods of time. Opened containers of diethyl ether should be checked periodically for peroxides using peroxide test strips.

3.6 The analyst should become familiar with the Material Safety Data Sheets (MSDS) for each reagent used in the extraction lab.

4.0 INTERFERENCES

4.1 Improperly cleaned glassware can contribute interferences to the sample extract. Material that is left inside the extraction vessels and glassware after cleaning may show up in the next extraction. The glassware must be properly cleaned according to SL SOP AN60.

4.2 Samples with high levels of organic material (oils, particulates, etc.) may cause the formation of emulsions during the extraction. Emulsions will occur most readily during the "base shake" to remove the non-target compounds. The extract may be filtered or stirred to remove the emulsion or may be "salted out" by the addition of sodium chloride.

4.3 The glassware used for herbicides should not be used to extract or concentrate dioxins and furans. Several of the herbicides are precursors to the formation of dioxins or are associated with the presence of dioxins in the environment

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 and an orange dot is affixed to the container to indicate this. The sample is stored at 4°C± 2°C in the lab. The sample must be extracted within seven (7) days of collection and the extract must be analyzed within forty (28) days of extraction. TCLP leachate samples are stored in glass containers at 4°C± 2°C from the TCLP to the extraction. The extraction must be performed within seven(7) days of the TCLP and the extract analyzed within forty(28) days of extraction

5.2 Soils, sediments, sludges, and wastes are collected in glass containers equipped with Teflon-lined caps. The normal container is 500-mL glass. Larger or smaller containers may be supplied. The sample is not preserved and should have an orange dot affixed to indicate this. The samples are stored at 4C ± 2C in the lab. The hold time for SVOC in solid matrices is 14 days from the date of collection. The extract must be analyzed within 28 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Separatory funnels - 1-L and 2-L, Teflon with Teflon stopcocks. Glass funnels may also be used.

6.2 Chromatography columns

6.3 Small funnels

6.4 Pyrex glass wool - purified by Soxhlet extraction with methylene chloride. The glass wool should be rinsed with acidified methanol prior to use.

6.5 Disposable, glass volumetric pipettes - 1.0-mL and 10-mL

6.6 Stainless steel spatulas

6.7 Wide range and narrow range pH paper

6.8 Pre-cleaned 500ml extraction bottles, P/N SAV-145. Discard after use.

6.9 1-L pre-cleaned containers. This is the same container used to collect the sample and can be used to collect the aqueous phase during cleanup. Discard after use.

6.10 Ultrasonic disrupter: Tekmar Model or equivalent with horn-type titanium-tipped sonication probe. The sonicator should be capable of operating in the pulse mode at full power.

6.11 Sonabox - the sonicator should be placed in the sonabox to reduce noise. The sonabox must be placed under a fume hood.

6.12 Filter paper-grade 414, 18.5cm diameter (VWR 28320-143)

6.13 Disposable volumetric pipets

6.14 Top-loading balance - capable of weighing to 0.1g. The balance calibration must be verified according to SL SOP AN10 each time the balance is used.

6.15 12mL vials with Teflon-lined caps.

6.16 Diazomethane generator

7.0 REAGENTS

7.1 Reagent water-lab generated deionized water

7.2 Sodium sulfate, powdered and granular, anhydrous - purified by heating at 400C for four (hours) in a shallow tray.

7.3 Acidified sodium sulfate-Transfer purified sodium sulfate to a suitable glass container until the container is about ¾ full. Working under a hood, add enough diethyl ether to wet the sodium sulfate. Add 1-2mL of concentrated sulfuric acid for each kilogram of sodium sulfate and stir to mix the acid into the solvent and sodium sulfate. Add more diethyl ether to keep the sodium sulfate wetted while stirring the sodium sulfate. The acid must be thoroughly mixed into the sodium sulfate and diethyl ether. Pour off the excess solvent and allow the ether to evaporate

under a hood for 24 hours. Store in a glass container. Check the pH of the acidified sodium sulfate by mixing 1g and 5mL of water in a small container. The pH of the solution should be less than 4.

7.4 Acetone - residue grade or better

7.5 1:1 acetone/methylene chloride: Mix equal volumes of acetone and methylene chloride in an appropriate glass container.

7.6 Methanol - residue grade or better

7.7 Diethyl ether-residue grade or better. Check periodically for formation of peroxides.

7.8 Sodium hydroxide (NaOH) - reagent grade

7.9 Sodium hydroxide solution (10N): Dissolve 400g of NaOH pellets into about 500mL of reagent water contained in a 2-L beaker on a magnetic stirrer. Add the NaOH in small portions, with constant stirring, to minimize the time it takes to dissolve the pellets. A good deal of heat will be generated as the NaOH dissolves. After all 400g has been added, carefully dilute to 1000mL with reagent water. Mix the solution thoroughly and transfer to a storage container. Do not store sodium hydroxide solution in volumetric glassware or in containers with ground glass joints.

7.10 Sulfuric acid (H_2SO_4) - concentrated reagent grade

7.11 Sulfuric acid solution (1:1 v/v): Slowly and carefully add 550mL of concentrated H_2SO_4 to 500mL of reagent water contained in a 2-L beaker on a magnetic stirrer. Add the acid in small portions with constant stirring to reduce the heat evolved when the acid and water are combined. Cool and transfer the solution to a labeled storage container.

7.12 Hydrochloric acid (HCl)-concentrated, reagent grade

7.13 Potassium hydroxide-reagent grade

7.14 Potassium hydroxide solution(37%)-weight 37g of KOH into a 100mL volumetric flask and dilute to volume with reagent water.

7.15 Acidified methanol (approximately 0.12N). Carefully add 5mL of concentrated HCl to 400mL of methanol in a 500mL volumetric flask. Dilute to volume with methanol. Larger volumes may be prepared.

7.16 Diazald (N-methyl-N-nitroso-toluenesulfonamide)-reagent grade

7.17 Carbitol-reagent grade or better

7.18 Silicic acid

7.19 Hexane-residue grade or better

7.20 Sodium chloride-reagent grade, purified, if necessary, by Soxhlet extraction with methylene chloride or by heating at 400C for four (hours) in a shallow tray.

8.0 STANDARDS

The preparation of the spiking solutions 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 expiration date of all spiking solutions is 40 days from date of 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 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Ci \otimes Vi = Cf \otimes Vf$$

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

where
Vi = volume of stock standard (or *initial standard*) needed to prepare the spiking solution(mL)
Ci = concentration of stock solution(or *initial standard*)(ug/mL)
Cf = concentration of spiking solution to prepare(*final concentration*)(ug/mL)
Vf = volume of spiking solution to prepare(*final volume*)(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 *Ci* and *Cf*.

8.2 Preparation of the Herbicide Surrogate Spiking Solution

8151 Surrogate Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
DCAA (Ultra PPS-165)	100	1.0	50	2.0

Solvent: Methanol

8.3 Preparation of the Herbicide Matrix Spiking Solutions

8151 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Chlorinated Herbicides Mixture (Ultra HBM-815A)	100	0.50	50	1.0

Solvent: Methanol

8151-TCLP Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Herbicide TCLP Mix (Restek 32014)	2000	0.025	50	1.0

Solvent: Methanol

8151-Full Target Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Chlorinated Herbicides Mixture 8150B(Ultra HBM-8150A)		1.0	20	
Dicamba, Silvex, 2,4,5-T	10			0.50
Dinoseb	50			2.5
2,4-D, Dichorprop, 2,4-DB	100			5.0
Dalapon	250			12.5
MCPA, MCPP	10000			500

Solvent: Methanol

9.0 SAMPLE PREPARATION

The samples and QC items must be clearly and unambiguously labeled during each step of the extraction and analysis process. Labeling tape should be used to identify the glassware associated with each sample. Solvents will erase grease pens and "Sharpies." Use of different colored labeling tape will aid the analyst in keeping up the extracts during each step of the extraction and concentration

9.1 Extraction of Aqueous Samples and TCLP Leachates

9.1.1 Remove the samples from the storage refrigerator and allow the sample to come to room temperature. Begin filling out the extraction log and gathering the required glassware while the samples are warming up. The glassware should be rinsed with acidified methanol and then with diethyl ether prior to extraction.

NOTE: All glassware used in this procedure must be rinsed with acidified methanol and diethyl ether prior to use.

9.1.2 Inspect the samples. Determine if the samples have multiple layers such as sediment or an oil layer. Sample with large amounts of sediments or particulates may clog the stopcock on the separatory funnel. Consult with the supervisor or technical manager if the sample matrix is unusual or is difficult to categorize or handle. Any unusual sample preparation steps required prior to the extraction must be noted on the extraction log or in an anomaly report.

9.1.3 Thoroughly mix the sample by inverting the container several times and pour 500mL of the sample container into a graduated cylinder that has been rinsed with acidified methanol. Transfer the sample to a properly labeled 1-L or 2-L Teflon separatory funnel. For TCLP samples, transfer 10mL of the leachate to a separatory funnel and dilute to 500mL with reagent water

9.1.4 Add 500mL of reagent water to each of two separatory funnels to serve as the method blank and lab spike (LCS).

9.1.5 Add 1.0 mL of surrogate to each sample, method blank, lab spike and matrix spike.

9.1.6 Add 1.0 mL of matrix spiking solution to all appropriate lab spikes and matrix spikes.

NOTE: Every extraction batch will have a method blank, a laboratory control spike (LCS), and a matrix spike and matrix spike duplicate (MS/MSD). The method blank and lab spike will be prepared using reagent grade water. An extraction batch is not to exceed 20 client samples. If insufficient sample is available for the MS/MSD, the extraction log must be stamped: " Insufficient

sample is available to perform the MS/MSD" and a lab control duplicate (LCSD) is extracted.

9.1.7 Add 100g of sodium chloride ot each sample and QC item.

9.1.8 Adjust the pH of the sample to >12 10N NaOH. Check the pH with wide range pH. Allow the samples to stand for at least one hour with intermittent shaking or shake on the automatic shaker for one hour.

NOTE: This step is necessary to convert the acid and ester forms of the herbicides to the water-soluble salts. After the hydrolysis step, the non-target compounds are extracted out of the sample with methylene chloride. The rest of the extraction steps may be performed with manual or automatic shaking.

9.1.9 Add 200 mL of diethyl ether to each separatory funnel.

9.1.10 Shake each separatory funnel for three minutes with periodic venting to release any excess pressure. If an automatic shaker is used, shake the samples for 10 minutes, Allow ten minutes for complete separation between the lower water and upper solvent phase.

NOTE: The separatory funnel should be vented under a hood to remove the solvent fumes from the lab.

9.1.11 Drain the lower water phase into a pre-cleaned 1-L container that has been rinsed with acidified methanol . The container must be large enough to hold more than 500mL because some of the diethyl ether will dissolve into the water. Discard the organic phase. If the sample contains high levels of organics, the solvent clean-up may be repeated two more times.

NOTE: Samples with high levels of organic material (oils, particulates, etc.) may cause the formation of emulsions during the extraction. Emulsions will occur most readily during the "base shake" to remove the non-target compounds. The extract may be filtered or stirred to remove the emulsion. Small aliquots of sodium sulfate may also be gently added to the extract to remove the emulsion.

9.1.12 Pour the sample back inot the separatory funnel and adjust the pH of each sample and the QC items to <2 with cold 1:1 sulfuric acid. Add the acid slowly and gently swirl the separatory funnels after the addition of the acid to ensure that the acid and base have reacted. Acid/base neutralization reactions can be violent if mixed too quickly. Check the pH of the samples and QC items to ensure that the pH < 2.

9.1.13 Add 200mL of diethyl ether to each sample and QC item.

-If performing a "manual shake", shake the funnels intermittently over a period of one hour, venting frequently to release any pressure.

-If using the automatic shaker, shake the samples continuously for 30 minutes, releasing the pressure periodically.

9.1.14 After the extraction, allow the layers to separate for at least 10 minutes. Collect the water layer in a large beaker or flask for disposal. Try and remove as much of the water as possible. Discard the water layer.

9.1.15 Collect the extract (upper layer) into a 500mL pre-cleaned extraction bottle containing 30g of acidified sodium sulfate. Allow the extract and sodium sulfate to remain in contact at least two

hours but preferably overnight. This is a good stopping point.

9.1.16 Place a piece of filter paper into a glass funnel and add a small amount of purified, acidified sodium sulfate. Filter the extract through the glass funnel and collect the extract directly into a labeled K-D apparatus.

9.1.17 The extract is concentrated to a final volume of approximately 10mL. The extract should be left in the 10mL graduated concentrator tube or transferred to a labeled storage vial. The extract is now ready for the diazomethane esterification.

9.2 *Extraction of Soils and Solids*

9.2.1 Remove samples to be extracted from the storage refrigerator and allow the samples to come to room temperature while the extraction glassware is being prepared. Begin filling out the extraction log and gathering the required glassware while the samples are warming up. The glassware should be rinsed with acidified methanol and then with diethyl ether prior to extraction. Check the "tune" of the sonicator using the procedure tested in SL SOP AN15: *Tekmar Sonic Dismembrator Tuning Procedure*.

9.2.2 Collect the appropriate glassware and rinse with acidified methanol and diethyl ether prior to use.

9.2.3 Prepare a number of funnels to filter the extracts by placing a piece of folded filter paper into the funnel. Rinse with acidified methanol and discard the solvent. Add about 2 - 3 inches of purified, acidified granular sodium sulfate to the filter paper in the glass funnel.

NOTE: Record as much information on the extraction log as possible before beginning the extraction to minimize the number of entries required during the extraction.

9.2.4 Open the sample container and inspect the sample. Note any unusual characteristics such as the presence of rocks, sticks, leaves, or other materials. Thoroughly homogenize the sample, mixing in any water that may be present on top of the sediment or soil. SL SOP AN70 gives guidance for *homogenization of samples*.

NOTE: If it is difficult to homogenize the sample or if the sample matrix is difficult to characterize, contact the supervisor before preceding with the extraction. A careful inspection of the sample at this point can save time and effort later on in the analysis. Any unusual sample preparation steps required prior to the extraction must be noted on the extraction log or in an anomaly report.

9.2.5 Weigh 30.0 - 30.5g of the homogenized sample into a pre-cleaned, labeled 500-mL extraction bottle. Record the weight for all samples in this batch.

NOTE: The lab has the option of weighing an aliquot of sample equal to 30g on a dry weight basis.

9.2.6 Weight 30g of powdered, purified sodium sulfate into two separate beakers to serve as the method blank and lab control sample(LCS).

9.2.7 Weigh two additional 30g portions of the sample in the batch selected as the matrix spike (MS) and matrix spike duplicate(MSD).

NOTE: Each extraction batch of twenty or fewer samples will have the following QC items: method blank, LCS, LCSD, MS, and MSD. If there is not enough sample to perform the MS/MSD, the extraction log must be stamped "INSUFFIECIENT SAMPLE WAS AVAILABLE TO PERFROM

THE MS/MSD" and the LCS extracted in duplicate(LCSD).

9.2.8 Working under a hood, acidify each sample and QC item with 0.1 to 0.2mL of concentrated hydrochloric acid. Add the acid to the sample slowly and carefully, stirring the sample with a stainless steel spatula or glass rod or pipette. Continue to add acid until the pH <2 when read with wide range paper.

9.2.9 Add powdered, acidified sodium sulfate to each sample and QC item. Stir with a spatula glass rod or stainless steel spatula to form a sandy, free-flowing mixture. The sodium sulfate combines with the water in the sample to "dry" the sample (remove the water). More sodium sulfate may be required if the sample is very wet. Precede to the next step as quickly as possible.

9.2.10 Add 1.0mL of the herbicide surrogate spiking solution to the method blank, LCS, MS, MSD and each sample in the batch.

9.2.11 Add 1.0ml of the appropriate herbicide matrix spiking solution to the LCS, MS, and MSD. Transfer the extraction bottle to a hood near the sonicator.

9.2.12 Under a hood, add 100mL of 1:1 acetone/methylene chloride to each sample and QC item. Stir the sample to break up any lumps that may have formed. Add more solvent until the solids are covered by about one inch.

9.2.13 Place the tip of the sonicator horn in the center of the beaker about ½ inch below the surface of the solvent but above the solid portion. More solvent may be added to bring the level to approximately one inch above the solid layer.

9.2.14 Sonicate for three (3) minutes with output control knob set at 10, mode switch to pulse, and percent duty cycle set at 50% If the sonication is properly performed, the solids and solvent will vigorously mix each time the sonicator pulses.

9.2.15 Under a hood, decant the extract through the filter funnel and collect the extract directly into the K-D apparatus. The extract may also be collected in a labled, pre-cleaned 500mL extraction bottle.

9.2.16 Repeat the extraction two more time two more times with fresh portions of solvent , collecting the solvent in the K-D apparatus or in the 500mL bottle each time.

NOTE: Clean the sonicator horn between samples by rinsing with acetone. "Sonicate" an extraction bottle of acetone/methylene chloride for 3 minutes with output control at 5 to further clean the horn (sodium sulfate may be added to the beaker to prevent cracking).

9.2.17 Concentrate the extract to approximately 10mL using the K-D concentration apparatus. The water bath should be 60-70C. Remove the K-D from the water bath and allow the extract to cool.

9.2.18 Remove the Snyder column and add 5mL of 37% KOH and 5mL of reagent water to to the extract in the K-D. Swirl the flask to mix. Replace the Snyder column and return the apparatus to the water bath set at 90-100C. Allow the K-D to remain on the water bath for a minimum of thirty minutes to hydrolyze the acid and ester forms of the herbicides to the acid form.

NOTE: The extract must reflux in the potassium hydroxide to hydrolize the herbicides to the salt form. The temperture of the water bath should be set to >90C in order to remove the residual solvent and allow the hydrolysis to occur. This temperture is higher than given in Method 8151A but is necessary for the proper reaction ot ocur.

9.2.19 Remove the K-D from the water bath and allow the apparatus to cool. Check the pH of the solution remaining in the K-D. The pH should be >12. If the pH is not greater than 12, add another 5mL of 37% KOH and 5mL o f reagent water and repeat step 9.2.18

NOTE: IF a small volume of solvent is present above the hydrolized herbicides (the aqueous layer), the solvent may be evaporated using a gentle stream of nitrogen.

9.2.20 Transfer the extract to a 1-L Teflon separatory funnel containing 500mL of reagent water. Use several small aliquots of reagent water to rinse the K-D body and the concentrator tube, adding the rinsings to the separatory funnel.

9.2.21 Swirl the funnel to mix the water and the basic solution from the K-D. The pH of the solution should be >12. If the pH in not greater than 12, add a small amount of 10N NaOH to adjust the pH >12.

9.2.22 Start at 9.1.8 and complete the cleanup and extraction of the herbicides in the same manner as the aqueous sample

9.3 Extraction of Wastes

9.3.1 Remove samples to be extracted from the storage refrigerator and allow the samples to come to room temperature while the extraction glassware is being prepared. Begin filling out the extraction log and gathering the required glassware while the samples are warming up. The glassware should be rinsed with acidified methanol and then with diethyl ether prior to extraction.

9.3.2 Prepare a number of funnels to filter the extracts by adding a small plug of glass wool that has been rinsed with acidified methanol and about 2 - 3 inches of purified, acidified granular sodium sulfate to the glass funnel.

NOTE: Record as much information on the extraction log as possible before beginning the extraction to minimize the number of entries required during the extraction.

9.3.3 Open the sample container and inspect the sample. Note any unusual characteristics such as the presence of rocks, sticks, leaves, or other materials. Thoroughly homogenize the sample.

NOTE: If it is difficult to homogenize the sample or if the sample matrix is difficult to characterize, contact the supervisor before preceding with the extraction. A careful inspection of the sample at this point can save time and effort later on in the analysis. Any unusual sample preparation steps required prior to the extraction must be noted on the extraction log or in an anomaly report.

9.3.4 Weigh 1-1.2g of the homogenized sample into a labeled, calibrated 12mL vial. Record the weight for all samples in this batch. Weigh two additional 1g aliquots of one sample to serve as the MS and MSD.

NOTE: Each extraction batch of twenty or fewer samples will have the following QC items: method blank, LCS, LCSD, MS, and MSD. If there is not enough sample to perform the MS/MSD, the extraction log must be stamped "INSUFFIECIENT SAMPLE WAS AVAILABLE TO PERFROM THE MS/MSD" and the LCS extracted in duplicate(LCSD).

9.3.5 Add 1.0mL of the herbicide surrogate spiking solution to the method blank, LCS, MS, MSD and each sample in the batch.

9.3.6 Add 1.0ml of the appropriate herbicide matrix spiking solution to the LCS, MS, and MSD.

9.3.7 Add diethyl ether to the sample until the volume comes up to the calibration line. Mix the sample, solvent, and surrogates and/or matrix spiking solutions by inverting the vials several times.

9.3.8 Add 500mL of reagent water to 1-L or 2-L separatory funnels and adjust the pH of the water to >12 with 10N NaOH. Label the separatory funnels and pour the extracts into the correspondong extration vessel. Check the pH with wide range pH and aadjust to pH > 12 if necessary with 10N NaOH. Allow the samples to stand for at least one hour with intermittent shaking or shake on the automatic shaker for one hour.

9.3.9 Starting at Section 9.1.8, continue the clean-up, extration, and estrification in the same manner as aqueous samples.

9.4 Esterification with Diazomethane

The extracts are contained in a 10mL graduated concentrator tube or should be transferred to 10mL graduated tubes prior to the esterification. The esterification of the extracts must be performed under a hood.

9.4.1 Concentrate the extracts to approximately 1mL under a gentle stream of nitrogen.

9.4.2 After all of the extracts have been concentrated, prepare the diazomethane generation device (Figure 1) for the esterification. Inspect the lines to ensure that there are no leaks or broken connections.

9.4.3 Place the extracts on the support. Replace all of the needles and place the needles into the first extracts to be esterified.

9.4.4 Add diethyl ether to the first container on the diazomethane generation device until the container is about ¾ full.

9.4.5 Add the following to the second container to esterify approximately 20 samples.

50mL diethyl ether
50mL 37% potassium hydroxide
50mL Carbitol
5g Diazald

Quickly attach the container to the diazomethane device and start the nitrogen flow. Recall that the needles should already be placed in the samples that are to be esterified first. The gas flow should be steady but not so high that the sample is bubbled out of the concentrator tube or that the sample is evaporated before the esterification can take place.

9.4.6 Allow the diazomethane to flow through the sample extracts until a persistent yellow color remains. This will usually take two to three minutes. As the rest of the samples are esterified, the process will take longer as the diazomethane is exhausted.

NOTE: For dark extracts where the persistent yellow color cannot be distinguished, esterify the sample for 10 minutes.

9.4.7 After the persistent yellow color remains, remove the needle from the tube in that position and replace it with a new needle. Place the needle into the next sample to esterify. Repeat for all samples in the batch, replacing the needle for each new extract.. If a yellow color cannot be formed in a clear extract, the diazomethane has most likely been exhausted. Pour the used reagents into a waste container (under a hood) and replenish the reagents in the second container. Add more diethyl ether to the first container if needed.

9.4.8 After all of extracts have been esterified, add a small amount (about 0.1g) of silicic acid to each sample extract, cover the concentrator tubes with aluminum foil and allow the extracts to sit for 30 minutes. The silicic acid will destroy any unreacted diazomethane. Dilute to 10mL with hexane and transfer the extract to a labeled storage vial. Store the extracts at 4C until the time of analysis.

10.0 ANALYTICAL PROCEDURE

No items in this revision. The analytical procedure is described in SL SOP SG65.

11.0 CALCULATIONS

No items in this revision.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Approval Signature: R. Wayne Robbins
Title: Corporate QA Manager Date : 8/8/97

ZYMARK EXTRACT CONCENTRATION PROCEDURE

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures for evaporating semivolatile organic extracts to final volumes appropriate to the analytical method. The procedures for "exchanging" a solvent, transferring the extract between containers, and the proper storage containers and conditions are included. This procedure is the preferred concentration preocedure fro maost analyses except chlorinated herbicides (SL SOP EX45) and dioxins and furans (SL SOP SM10).

2.0 SUMMARY OF METHOD

After the extraction procedure is completed, the solvent is transferred to a glass Zymark concentration tube. The tube is placed in the Zymark concentration device that has been heated to a specified temperature. A stream of nitrogen is directed into the tube to evaporate the solvent and to concentrate the target compounds. When the volume of solvent reaches the specified volume-normally 1mL-, the nitrogen is automatically stopped. An alarm sounds to alert the analyst and the extract is removed from the device and transferred to a storage vial or container. If a solvent exchange is required, the exchange solvent is added to the tube and the solvent is evaporated to the specified final volume and transferred to a storage vial or container. The concentrated extracts are stored at 4C until the time of analysis by GC, GC/MS, or LC.

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 you or others in potentially hazardous situations.

3.2 This SOP contains procedures that are designed to reduce the exposure of lab personnel to solvent vapors and to minimize the amount of solvent introduced into the lab air. All solvent transfer steps should be performed quickly and under a hood, if possible. Use the minimum amount of solvent to get the job done. Do not allow open containers of solvent or extracts to evaporate into the lab.

3.3 A lab coat or apron should be worn to protect clothing and skin from acids, bases, and solvents. Gloves must be worn to protect hands and eye protection (goggles or safety glasses) must be worn at all times in the lab. An analyst must not work alone in an isolated area.

3.4 A solvent such as methylene chloride can cause a burning sensation if it contacts the skin. The rapid evaporation of the solvent causes a rapid heat loss in the skin similar to frost bite. If solvent contracts the skin, wash the exposed skin in cold water to reduce the solvent evaporation.

3.5 The analyst should become familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in the extraction lab.

4.0 INTERFERENCES

Improperly cleaned Zymark tubes can contribute iartifacts to the sample extract. Material that is left inside the tube after cleaning may show up in the next extraction-this is especially true of extracts with high levels of target or non-target compounds. The glassware must be properly cleaned according to SL SOP AN60. After cleaning, inspect the glassware for the presence of water, especially in the small tip of the Zymark tube. A thorough acetone rinsing will help to eliminate or minimize this problem.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Concentrated extracts must be stored at 4C until the time of analysis. The volume of the extract must be accurately known and recorded.

6.0 APPARATUS AND MATERIALS

6.1 Zymark Turbo II concentration device or equivalent. The instrument must be vented into an operating fume hood.

6.2 Concentration tube-200 mL(for liquids) or 225mL elongated tubes(for soils) with 1.0mL tip.

6.3 Pasteur Pipettes- 1mL disposable

6.4 2-mL vials with Teflon lined crimp caps-the vials must be compatible with the autosampler

6.5 Storage vials- 12 mL glass with Teflon-lined caps. A storage vial may be calibrated by adding a known volume of liquid to the container and marking the volume on the side of the container.

6.6 Glass syringe- 10 ml

6.7 0.45 micron filters

6.8 11mm crimp-type capper and decapper

7.0 REAGENTS

7.1 Methylene Chloride

7.2 Hexane

7.3 Acetonitrile

7.4 Acetone

8.0 STANDARDS

This section is not applcable to this SOP.

9.0 SAMPLE (EXTRACT) PREPARATION

The sample and QC item extracts must be clearly and unambiguously labeled during each step of the concentration process. Labeling tape should be used to identify the glassware associated with each sample. Solvents will erase grease pens and "Sharpies."

Use of different colored labeling tape will aid the analyst in keeping up with the extracts during each step of the extraction and concentration.

9.1 Equipment Preparation

9.1.1 Pre-rinse each Zymark concentration tube with acetone.

9.1.2 Turn on evaporation unit. Set the water bath to the temperature that has been developed for the target compounds. The gas pressure should be set initially to zero for each cell in the Zymark unit. Table 1 provides guidance on the temperatures and pressures that can be used to successfully recover the target compounds.

9.1.3 Pre-rinse each concentration cap on evaporation unit with acetone.

9.2 Solvent Evaporation

9.2.1 f the sample extracts have been collected in any container other that a Zymark tube, the extract is transferred to a Zymark tube.Working under a hood, pour 150mL-200mL of extract (depending on the size of the tube) into labeled Zymark concentration tube and place the tube into a cell of the evaporation unit. Repeat for all samples in analytical batch. The remaining extract must be tightly covered with a piece of aluminum foil to minimize evaporation of the solvent into the lab.

NOTE: Most extracts will be dried and filtered directly into the Zymark tube.

9.2.2 Carefully close the cover of the Zymark unit completely. Make sure that each tube is seated properly and that the individual covers are direclty over each tube. When the elongated tubes are used, care must be taken in positioning the tubes in order to avoid breaking the tubes covers and to completely close the instrumetn cover.

9.2.3 Turn on the pressure for each unit and gradually increase the pressure to the maximum level. Start at a low pressure and gradually work up to the maximum. Table 1 provides guidance on the temperatures and pressures that can be used to successfully recover the target compounds.

9.2.4 Periodically add more sample until all of the sample has been transferred to Zymark concentration tube. Continue concentration until cell alarm sounds. This indicates the sample is at a 1mL volume.

9.2.5 If the solvent must be exchanged, add 25mL of the exchange solvent and set the Zymark to the conditions optimized for that solvent and the target compounds. Table 1 provides guidance on the temperatures and pressures that can be used to successfully recover the target compounds. Continue concentration until cell alarm sounds. This indicates the sample is at a 1mL volume.

Add a second 25mL aliquot of the exchange solvent to the Zymark tube and continue concentration until cell alarm sounds.

9.2.6 Remove Zymark concentration tube from evaporation unit.

9.3 Transferring Extracts to Storage Vials

9.3.1 Under a hood, adjust the volume to 1mL by adding small aliquots of the required solvent. If the final volume is to be greater than 1mL, add an appropriate volume of solvent to the Zymark tube using a volumetric pipette or calibrated solvent dispensing device. For

example, if the final volume is to be 10mL, adjust the volume in the tube to 1mL and then add 9mL of solvent to give a final volume of 10mL.

NOTE: If the extract cannot be evaporated to 1mL, the extract should be transferred to a volumetric flask or to a calibrated storage vial. Use several small aliquots of solvent to transfer the entire extract to the vial.

9.3.2 Under a hood, transfer the extract to aTeflon lined crimp top or 12 ml scintillation vial. Mark the volume of the extract on the side of the container to allow the analysts to judge whether the sample extract has evaporated during storage and handling. Store concentrated extracts at 4 C until time of analysis..

10.0 ANALYTICAL PROCEDURE

11.0 CALCULATIONS

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 Sample ID, extraction log, and transferring extracts to groups

12.2 Accurate final volumes marked on outside of container

13.0 PREVENTIVE MAINTENANCE

Change the water in the water bath weekly. Add 1-2 drops of XXXX Bath to prevent bacteria and algae growth. Methlyene chloride that dissolves in the water bath will damage the sensors.

14.0 TROUBLE-SHOOTING

Occasionally, the sensors have difficulty detecting the final volume of the solvent in the Zymark tubes. Refer to the manufacturer's manual fro procedures for testing the sensors and replacing them, if necessary.

15.0 REFERENCES

Table 1-ROUTINE EXTRACTS

METHODS	TYPES OF COMPOUNDS	ZYMARK TEMPERATURE	ZYMARK MAX PRESSURE	SOLVENT	FINAL SOLVENT	FINAL VOLUME(mL)
604,8041	Phenols by GC-FID	40 C	15psi	MeCl2	MeCl2	1.0
606,8061	Phthalate esters by GC-FID	40 C	15psi	Mecl2	MeCl2	1.0
607,8070	Nitrosamines by GC-FID	40 C	15psi	MeCl2	MeCl2	1.0
608,8080,8081	Chlorinated pesticides and PCBs	40 C	15psi	MeCl2	Hexane	10
609,8090	Nitroaromatics by GC-EC (2,4-DNT and 2,6-DNT)	40 C	15psi	MeCl2	Hexane	10
610,8100	PAH by GC-FID	40 C	15psi	MeCl2	MeCl2	1.0
611,8110	Halogenated ethers by GC/EC	40 C	15psi	MeCl2	MeCl2	1.0
612,8120	Chlorinated hydrocarbons by GC-EC	40 C	15psi	MeCl2	Hexane	10
614,614.1,622, 622.1,8141	Phosphorous and nitrogen containing pesticides	40 C	15psi	MeCl2	Hexane	10
617,618	Chlorinated pesticides	40C	15psi	MeCl2	Hexane	10
619	Phosphorous and nitrogen containing pesticides	40C	15psi	MeCl2	Hexane	10
625	BNA	40 C	15psi	MeCl2	MeCl2	1.0
8270-TCL,K001	BNA	40 C	15psi	MeCl2	MeCl2	1.0
8270-TCLP, AP9	BNA	40 C	15psi	MeCl2	MeCl2	1.0
8015-EXT	Petroleum products	40 C	15psi	MeCl2	MeCl2	1.0
8310	PAH by HPLC	40 C	15psi	MeCL2	Acetonitrile	1.0

Table 2-Extracts That Have Been Subjected to GPC Clean-Up

METHODS	TYPES OF COMPOUNDS	ZYMARK TEMPERATURE	ZYMARK MAX PRESSURE	SOLVENT	FINAL SOLVENT	FINAL VOLUME (mL)
608,8080,8081	Chlorinated pesticides and PCBs	40 C / 50 C	8psi	MeCl2	Hexane	5.0
8270-TCL,K001	BNA	40 C	8psi	MeCl2	MeCl2	0.50
8270-TCLP, AP9	BNA	40 C	8psi	MeCl2	MeCl2	0.50

Approval Signature: R. Wayne Robbins
Title: Corporate QA Manager Date: 11/25/97

PREPARATION OF SEMIVOLATILE SURROGATE AND MATRIX SPIKING SOLUTIONS

1.0 SCOPE AND APPLICATION

This SOP describes the procedures for preparing the surrogate and matrix spiking solutions used in the extraction of semivolatile compounds in aqueous and solids matrices.

A surrogate is a compound that is not expected to be present in an environmental sample. One or more surrogates are added to the sample prior to extraction as a check on the efficiency of the entire preparation process. Surrogates are usually brominated, fluorinated, or isotopically labeled compounds.

The matrix spiking solution contains the target compounds or a subset of the target compounds. This solution is added to a "blank" matrix (the lab control sample-LCS) to verify that the target compounds can be recovered within statistically defined limits in the absence of interferences. The matrix spiking solution is also added to aliquots of field sample to evaluate the effect that the sample matrix has on the recovery of the target compounds.

2.0 SUMMARY OF METHOD

Known volumes of certified stock standard solutions are diluted with an appropriate solvent in Class A volumetric glassware. Acetone and methanol are generally used as the solvents for the surrogates and matrix spiking solutions because they are miscible with water (that is, these solvents mix with water in all proportions). The tables included in this SOP describe the "recipes" that can be used to prepare the spiking solutions. The lab can prepare larger or smaller volumes if desired. The recipe used by the lab may be prepared as a "controlled posting" to prompt the analyst in the preparation of the spiking solutions. The controlled posting can be posted in the lab or placed in the extraction lab tracking log.

Table	Group	Methods
1	SM	625, 8270, CLP 3/90, CLP 10/92
2	SG-FID	604, 8040, 606, 8060, 610, 8100, 8015-modfied
3	SG-pesticides	608, 8080, 8081, CLP 3/90, CLP 10/92
4	SG-herbicides	615, 8151
5	SG-NPD	8141
6	LC	8310

NOTE: These tables list methods and spiking solutions that are common to two or more SL divisions. Some methods are only performed at one division and the preparation of the spiking solutions for those methods will be in the SL SOP for that 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 that will put you or others in potentially dangerous situations.

3.2 The analyst should wear a lab coat or apron, gloves, and protective lab glasses when preparing the spiking solutions. Work 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 INTERFERENCES

No items in this section for this revision of the SOP.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

No items in this section for this revision of the SOP.

6.0 APPARATUS AND MATERIALS

6.1 Volumetric flasks-Class A glass, assorted volumes. Recommended volumes: 5-,10-,25-, 50-, 100mL

6.2 Glass syringes and microsyringes-assorted volumes. Recommended volumes: 0,25,50,100uL, 1-,2.5-,5mL

6.3 Disposable volumetric pipets-1ml and 10mL

6.4 Storage vials with Teflon-lined caps. 40mL VOA vials or other storage vials

7.0 REAGENTS

7.1 Acetone-residue grade or equivalent

7.2 Methanol-residue grade or equivalent

7.3 Carbon disulfide-residue grade or equivalent.

8.0 STANDARDS

The preparation of the spiking solutions 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 expiration date of all spiking solutions is 40 days from date of 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 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Ci \otimes Vi = Cf \otimes Vf$$

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

where

Vi = volume of stock standard (or *initial standard*) needed to prepare the spiking solution(mL)
Ci = concentration of stock solution(or *initial standard*)(ug/mL)
Cf = concentration of spiking solution to prepare(*final concentration*)(ug/mL)
Vf = volume of spiking solution to prepare(*final volume*)(mL)

The concentration can be expressed in whatever terms the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for *Ci* and *Cf*.

EXAMPLE:

Prepare 50mL of the 625/8270 surrogate spiking solution from the 5000ug/mL base/neutrals and 10000ug/mL organic acids surrogates stock standards. The 625/8270 surrogate spiking solution has the base/neutrals at 50ug/mL and the acids at 100ug/mL.

Base/neutrals

$$Vi = \frac{Cf \otimes Vf}{Ci} = \frac{50ug/mL \otimes 50mL}{5000ug/mL} = 0.50mL$$

Acids

$$Vi = \frac{Cf \otimes Vf}{Ci} = \frac{100ug/mL \otimes 50mL}{10000ug/mL} = 0.50mL$$

0.50mL of the base/neutrals stock and 0.50mL of the acids surrogates stock are combined into a 50mL volumetric and diluted to a final volume of 50mL with acetone or methanol.

8.2 Basic Spiking Solution Preparation Steps

8.2.1 Remove the stock solutions from the refrigerator and allow the stock solutions to come to room temperature.

8.2.2 Prepare the volumetric glassware in accordance with SL SOP AN60. Rinse the flask thoroughly with the solvent that is used to prepare the spiking solution. Several small aliquots of the solvent will generally clean the flask better than one large aliquot and will minimize the amount and exposure to the of solvent

8.2.3 Fill the flask approximately half full with acetone or methanol.

8.2.4 Carefully open the ampoule or storage vial of the stock standard to be used to prepare the spiking solution and transfer the appropriate volume of the stock standard to the volumetric flask. The volume of standard required is calculated as in Section 8.1 or is found in the preparation recipe in the tables. Rinse the inside walls of the flask after each addition of stock standard to ensure that all of the standard is transferred to the flask. Repeat for each component of the spiking solution.

8.2.5 Dilute to volume with the appropriate solvent, cap the flask, and invert to mix the spiking solution. Transfer the spiking solution to a labeled storage vial. Transfer a 1mL aliquot to an autosampler vial for assay by the analytical group.

NOTE: If the standard is to be assayed by GC or GC/MS, the spiking standard solution may be stored temporarily in the volumetric flask to prevent the use of the standard until verified.

8.2.6 The standard traceability logbook must be filled out in accordance with SL SOP AN41. The spiking solution must be clearly labeled with the following information:

-standard identification or designation
-assigned SL lot number
-date prepared
-analyst's initials
-expiration date
-solvent

The volume of the solution must be marked on the side of the storage container so that the analysts can gauge whether evaporation or degradation has taken place. The volume should be marked at room temperature initially and after each aliquot is removed from the vial.

9.0 SAMPLE PREPARATION

No items in this section for this revision of the SOP.

10.0 PROCEDURE

No items in this section for this revision of the SOP. Procedure is described in Section 8.

11.0 DATA ANALYSIS/CALCULATIONS

No items in this section for this revision of the SOP.

12.0 QUALITY ASSURANCE/QUALITY CONTROL

12.1 All standards received into the lab and prepare by the lab must be tracked in accordance with SL SOP AN41:*Standard Material Traceability.* Each standard must be clearly labeled.

12.2 The temperature of the refrigerated standard storage areas must be monitored daily.

12.3 If the volumetric glassware is not Class A, the volumetric glassware must be verified to be within 1% of its stated volume or removed from service. See SL SOP AN30: *Pipette and non-Class A Volumetric Container Calibration Check*

13.0 PREVENTIVE MAINTENANCE

No items in this section for this revision of the SOP.

14.0 TROUBLESHOOTING

No items in this section for this revision of the SOP.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*, current revisions.

15.2 SW-846 Method 8000B

TABLE 1-SEMIVOLATILE GC/MS (SM)
Methods 625, 8270, CLP 3/90, CLP 10/92

625/8270 Surrogate Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Base/neutrals Surrogates (NSI C-376H)	5000	1.0	100	50
Acid Surrogates (NSI C-131H)	10000	1.0		100

Solvent: Acetone

8270 Matrix Spiking Solution
This spiking solution contains a subset of the 8270 compounds.

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Base/neutrals Matrix Spike Stock (Restek 31074	5000	1.0	100	50
Acid Matrix Spike Stock (Restek 31071)	10000	1.0		100

Solvent: Acetone

K001 (8270) Matrix Spiking Solution
This spiking solution contains phenols and PAHs that are associated with the K001 analysis by GC/MS.

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
SV Mix #2(Phenols) (Restek 31008)	2000	1.0	20	100
SV Mix #5 (PAH) (Restek 31011)	2000	1.0		100
2,3,4,6-Tetrachlorophenol (Restek 58-90-2)	1000	2.0		100
Carbazole (Restek 86-74-8)	1000	2.0		100

Solvent: Methanol

8270-TCLP Matrix Spiking Solution
This spiking solution contains a subset of the 8270 compounds that is used when a matrix spike is requested on a TCLP leachate.

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
TCLP B/N Mix (Restek 31028)	2000	1.0	20	1000
TCLP Acid Mix (Restek 31027)	2000	1.0		100

Solvent: Acetone

TABLE 1-SEMIVOLATILE GC/MS (SM)

625 Matrix Spiking Solution
This spiking solution contains all of the 625 compounds except the multi-peak target analytes. This solution must be prepared from stock solutions that are independent of the stocks used to prepare the calibration standards. This is the default matrix spiking solution. If a subset of the 625 target compounds is to be reported, a spiking solution containing only the subset of target compounds may be prepared.

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Method 604 - Phenols Cal Mix (Restek 31029)	2000	1.0	20	100
Method 606 - Phthalate Esters (Restek 31031)	2000			100
Method 609 - Nitroaromatics/ISO (Restek 31033)	2000			100
Method 610 - PAH (SV #5) (Restek 31011)	2000			100
Method 611 - Haloethers (Restek 31034)	2000			100
Method 612 - Chlorinated Herbs (Restek 31035)	2000			100
Method 605 - Benzidine (Restek 31030)	3000			100

Solvent: Acetone or methanol

NOTE: The individual solutions can be used to prepare the full target spiking solutions for the 600-series methods.

TABLE 1-SEMIVOLATILE GC/MS (SM)

CLP SPIKING STANDARDS

BCLP 3/90 Surrogate Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
B/N Surrogate Mix (NSI C-162-05)	5000	1.0	50	100
Acid Surrogate Mix (NSI C-191-03)	7500	1.0		150

Solvent: Acetone

BCLP 3/90 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
B/N Matrix Spike (3/90) (Restek	5000	1.0	50	100
Acid Matrix Spike (Restek	10000	0.75		150

Solvent: Acetone

BCLP 10/92 Surrogate Spiking Solution ('low level")

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
B/N Surrogate Mix (Restek	5000	0.40	50	40
L/C Acid Mix (Restek	3000	2.0		120

Solvent: Acetone

BCLP 10/92 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
L/C BN LCS A (Restek 31241A)	1000	0.50	25	20
L/C BN LCS B (Restek 21241B)	2000	0.50		40
LC Acid LCS (Restek 31212)	2000	0.50		40

Solvent: Acetone

TABLE 2-SEMIVOLATILE GC (SG) - FID METHODS
Methods 6004,8040,606,8060,610,8100,8015-modified

SG Acid Surrogate Spiking Solution
This spiking solution is used for Methods 604 and 8040.

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
8040 Surrogate Mix (Restek 31090)	2000	1.0	20	100

Solvent: Methanol

8040 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Acid Matrix Spike Stock (Restek 31071)	10000	0.50	50	100

Solvent: Methanol

604 Matrix Spiking Solution
This spiking solution contains all of the 604 compounds. This solution must be prepared from stock solutions that are independent of the stocks used to prepare the calibration standards. This is the default matrix spiking solution. If a subset of the 604 target compounds is to be reported, a spiking solution containing only the subset of target compounds may be prepared.

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Method 604-Phenols Cal Mix (Restek 31029)	2000	1.0	20	100

Solvent: Methanol

SG Base Surrogate Spiking Solution
This spiking solution is used for Methods 606, 8060, 607,8070,610, 8100, and 8015-Modified

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
2-Fluorobiphenyl (Restek 31091)	2000	1.25	50	50
o-Terphenyl (Restek 31066)	2000	1.25		

Solvent: Acetone or methanol

606/8060 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Method 606 - Phthalate Esters (Restek 31031)	2000	0.5	20	50

Solvent: Acetone or methanol

TABLE 2-SEMIVOLATILE GC (SG) - FID METHODS

610 Matrix Spiking Solution
This spiking solution contains all of the target analytes. This solution must be prepared from a stock that is independent of the stocks used to prepare the calibration standards

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Method 610 - PAH (SV #5) (Restek 31011)	2000	0.50	20	50

Solvent: Acetone or methanol

8100 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
PAH Custom Mix (Restek	2000	0.5	20	50

Solvent: Acetone or methanol

Diesel-8015-Modified

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
XHc Diesel Fuel #2 Composite (Restek 31259)	50000	1.0	50	1000 (1.0 mg/mL)

Solvent: Acetone

Mineral Spitrits-8015-Modified

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
XHc Mineral Spirits Standard (Restek 31261)	50000	1.0	50	1000 (1.0 mg/mL)

Solvent: Acetone

Florida PRO Surrogate Spike

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
o-Terphenyl (Restek 31066)	2000	5.0	200	50
C-39 Hydrocarbon Std (NSI 1339-04-01)	6000	5.0		150

Solvent: 1:1 Acetone/Carbon disulfide

Florida PRO Matrix Spike

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
C8-C40 Hydrocarbon Std (NSI C-443-03)	2000	2.5	100	50

Solvent: 1:1 Acetone/Carbon disulfide

TABLE 3-SEMIVOLATILE GC (SG) - PESTICIDES
Methods 608,8080,8081, CLP 3/90, CLP 10/92

Pesticides Surrogates Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Pesticides Surrogate Mix (Restek 32000)	200	0.100	100	0.20

Solvent: Acetone

8080 Pesticides Matrix Spiking Solution
This spiking solution contains a subset of the target analytes.

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Pesticides Matrix Spike Mix (2/88) (Restek 32031)	0.200	0.050	50	0.2

Solvent: Acetone

608 Pesticides Matrix Spiking Solution
This solution contains all of the single peaks pesticides. This solution must be prepared from a stock that is independent of the stock used to prepare the calibration standards.

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Method 608 Calibration Mix (Restek 32022)	200	0.10	100	0.20

Solvent: Acetone

PCB Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Ar1016 (Restek 32006)	1000	0.125	50	2.5
Ar1260 (Restek 32012)	1000	0.125		2.5

Solvent: Acetone

TCLP-Pest

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
TCLP Pesticide Mix (Restek 32013)	2000	0.010	100	0.20

Solvent:acetone

TABLE 3-SEMIVOLATILE GC (SG) - PESTICIDES

CLP SPIKING STANDARDS

PCLP 3/90 Surrogate Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Pesticide Surrogate Mix (Restek 32000)	200	0.100	100	0.20

Solvent: Acetone

PCLP 3/90 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
3/90 Pesticide Matrix Spike (Restek 32018)	25/50	1.0	50	0.50/1.0

Solvent: Acetone

PCLP 10/92 Surrogate Spiking Solution ('low level")

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Pesticide Surrogate Mix (Restek 32000)	200	0.10	50	0.040

Solvent: Acetone

PCLP 10/92 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
L/C Pesticide Lab Control Sample (Restek 32040)	10-20	0.10	25	0.04-0.08

Solvent: Acetone

TABLE 4-Semivolatile GC (SG) Herbicides
Methods 8151

8151 Surrogate Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
DCAA (Ultra PPS-165)	100	1.0	50	2.0

Solvent: Methanol

8151 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Chlorinated Herbicides Mixture (Ultra HBM-815A)	100	0.50	50	1.0

Solvent: Methanol

8151-TCLP Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Herbicide TCLP Mix (Restek 32014)	2000	0.025	50	1.0

Solvent: Methanol

8151-Full Target Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Chlorinated Herbicides Mixture 8150B(Ultra HBM-8150A)		1.0	20	
Dicamba, Silvex, 2,4,5-T	10			0.50
Dinoseb	50			2.5
2,4-D, Dichorprop, 2,4-DB	100			5.0
Dalapon	250			12.5
MCPA, MCPP	10000			500

Solvent: Methanol

TABLE 5-Semivolatile GC (SG) Nitrogen/Phosphorus Detector(NPD)
Methods 8140/8141

8141 Surrogate Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Triphenyl phosphate (EM Science)	1000	0.20	50	4.0

Solvent: Acetone

8141 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
Thionazin (Supelco)	2000	0.125	50	5.0
Diazinon (Accustandard)	1000	0.250		5.0
Ronnel (Accustandard)	100	2.5		5.0
Ethyl parathion (Accustandard)	100	2.5		5.0
Methyl parathion (Accustandard	100	2.5		5.0

Solvent: Acetone

TABLE 6-Semivolatile Liquid Chromatography (LC)
Method 8310

8310 Surrogate Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
4-Terphenyl-d14 (Supelco 4-8418)	2000	0.125	100	2.5

Solvent: Acetone

8310 Matrix Spiking Solution

Stock Standards	*Cstock* (ug/mL)	*Vstock* (mL)	*Vspike* (mL)	*Cspike* (ug/mL)
TCL PAH (Supelco 4-9156)	20-1000	0.250	50	0.10 - 5.0

Solvent: Acetone

Approval Signature: R. Wayne Robbins

R. Wayne Robbins

Title: Corporate QA Manager Date: 8/29/97

TOTAL ORGANIC CARBON IN SOIL: WALKLEY-BLACK METHOD

1.0 SCOPE AND APPLICATION

1.1 This method is used to estimate the concentration of organic carbon in soils, sludges, and oils. The carbon in carbonized materials such as charcoal is not amenable to this procedure.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits are given in Section 5 of the Savannah Laboratories Quality Assurance Plans.

2.0 SUMMARY OF METHOD

2.1 Organic carbon in soils is determined by adding 10 mL of standard potassium dichromate and sulfuric acid to a weighed soil sample. After allowing sufficient time for oxidation of the organic carbon, the remaining dichromate is back-titrated with standardized ferrous sulfate and the organic carbon is estimated.

2.2 This method is based on Method 29-3.5.2 from *Methods of Soil Analysis*(1).

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 you or others in potentially dangerous situations.

3.2 This procedure calls for the use of a strong oxidizer (potassium dichromate) and a strong acid (sulfuric acid). The analyst must wear a lab coat or apron, protective eye glasses or face shield, and gloves when performing this procedure.

3.3 Material Safety Data Sheets (MSDS) are available for each reagent and standard used in the lab. The analyst should be thoroughly familiar with the MSDS which list safe handling and storage parameters for the materials.

4.0 INTERFERENCES

4.1 The organic matter in soils is, in general, not quantitatively oxidized by the dichromate in this procedure. The TOC result obtained is therefore multiplied by a correction factor to account for the generally low recoveries obtained by the method.

4.2 Certain oxidizable or oxidizing inorganic elements or compounds may interfere. High concentrations of chloride can be a significant positive interference. If the presence of a high level of chloride is suspected, the following correction equation can be used:

TOC, mg/L = Apparent TOC, mg/L - (mg/L Cl^-/12).

When Fe^{2+} is present in soil, its oxidation by dichromate will result in a positive interference. Iron is generally in the oxidized state (Fe^{3+}) in aerated soils, and thus, this generally is not a problem. Highly reduced soil types, such as deeply buried, fine-grained sediments, may contain appreciable quantities of Fe^{2+}. These samples should be dried in a drying oven at 105°C prior to analysis. This will allow for the Fe^{2+} to be oxidized to Fe^{3+}. MnO_2 present can be a negative interference, but only freshly precipitated MnO_2 takes part in this redox reaction; thus MnO_2 interference is thought not to be a serious error in most soils (1).

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Sampling and storage of samples in glass bottles is preferable. Use of conventional polyethylene containers is permissible if it is established that the containers do not contribute contaminating organics to the sample. The sample should be stored at 4C ± 2C until analysis. The analysis should be performed as soon as possible but should be completed within 28 days of collection.

6.0 APPARATUS AND MATERIALS

6.1 Analytical balance

6.2 Drying oven

6.3 Buret, 25-mL

6.4 Erlenmeyer flasks, 250-mL

7.0 REAGENTS

The preparation and standardization of all reagent must be documented in the appropriate log.

7.1 Reagent water - lab generated deionized water

7.2 Potassium dichromate, 1 N: Dissolve 49.04 g of reagent grade $K_2Cr_2O_7$ (dried at 105°C) in DI water in a 1-L volumetric flask, and dilute to volume.

7.3 Ferrous sulfate heptahydrate, 0.5 N: Dissolve 140 g of reagent grade $FeSO_4 \cdot 7H_2O$ in DI water, add 15 mL concentrated sulfuric acid, and dilute to 1000 mL. This titrant should be standardized with each set of samples.

Calculate the normality of the ferrous sulfate as shown below:

$$[FeSO_4], N = \frac{10\ mL \times 1\ N}{mL\ FeSO_4\ used\ for\ titration}$$

Standardize ferrous sulfate daily by titrating 10 mL of potassium dichromate solution.

7.4 Sulfuric acid, concentrated

7.5 Ferroin indicator: Commercially available 0.025M o-phenanthroline-ferrous complex

8.0 STANDARDS

No calibration standards are associated with this procedure. A check standard may be obtained from a reputable vendor such as ERA, APG, or ASI. Municipal digest sludge may be used as a check standard if a certified value is supplied. Ten milliliters (1mL) of the 2000mg/L TOC standard may be also used as a check standard (assume the sample weight is 10g).

8.1 Potassium hydrogen phthalate (KHP)-reagent grade or better.

8.2 Potassium hydrogen phthalate, 2000mg Carbon / liter
Dissolve 0.4256g of reagent grade KHP in approximately 800mL of reagent water contained in a 1.0-L volumetric flask. Dilute to volume with reagent water and transfer the stock standard to an amber glass container. Label the container with the standard identification, concentration, analyst's initials, date prepared, and the expiration date. Record the preparation in the standards prep log. Store the standard in the refrigerator at 4C +/-2C. Prepare this standard monthly or as needed.

9.0 SAMPLE PREPARATION

The sample preparation steps are included as part of the Procedure (Section 10).

10.0 PROCEDURE

10.1 Remove the sample from the refrigerator and allow the samples to come to room temperature. Thoroughly mis the material, removing sticks, rocks, and leaves.

10.2 Weigh out soil sample to contain roughly 10 to 25 mg organic C into a 250-mL Erlenmeyer flask.

- Dark soils and sludges generally contain high concentrations of organic carbon and 0.05 - 0.1 g is weighed out for these samples.
- For medium-colored soils, 0.1 - 2.5 g of sample is used.
- For light-colored soils and sands, 1-5 g of sample is weighed out.

10.3 Place Erlenmeyer flasks containing saturated sediments or other possibly anaerobic soils into a drying oven at 105°C for 1 h, or until they are thoroughly dry.

10.4 Add 10 mL potassium dichromate solution by pipet and swirl to mix. Rapidly add 20 mL of concentrated H_2SO_4 (under a ventilating hood) and swirl to mix. Allow to cool and add 150 mL DI water.

10.5 Filter mixture through a glass fiber filter if it is too turbid to see color changes.

10.6 Add 8-10 drops ferroin indicator. Titrate with ferrous sulfate until color changes from blue-green to maroon. If the titration requires less than 5 mL of $FeSO_4$ titrant, repeat the analysis with a smaller quantity of soil.

11.0 CALCULATIONS

Calculate sample results with the following formula:

$$Organic\ C, mg/kg = \frac{[(mL\ K_2Cr_2O_7 \times 1\ N) - (mL\ FeSO_4 \times FeSO_4)] \times 3000 \times f}{(W)(solids)}$$

where

W = weight of soil (g)
solids = (percent solids)/100
f = 1.3

The factor f is empirically determined, but is taken to have a value of 1.3.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 Ferrous sulfate is standardized daily by titrating it against 10 mL of 1 N $K_2Cr_2O_7$.

12.2 A QC check standard, municipal digested sludge, or 2000mg/L TOC standard is analyzed in duplicate for every twenty samples.

13.0 TROUBLESHOOTING

No items are included in this revision.

14.0 PREVENTIVE MAINTENANCE

No items are included in this revision.

15.0 REFERENCES

15.1 *Methods of Soil Analysis*; Page, A.L., *et.al.*, ed. American Society of Agronomy: Madison, WI, 1982.

15.2 *Savannah Laboratories' Comprehensive Quality Assurance Plan* and *Savannah Laboratories' Corporate Quality Assurance Plan*, current revisions.

TOTAL ORGANIC CARBON
WALKLEY-BLACK METHOD

Date Received	Analyst's Initials	Sample Log	Sample I.D.	Sample Weight	Titer mL	TOC (mg/kg)	Dry Weight	TOC dwb

BA09:05.01.98:1

Approval Signature:	R. Wayne Robbins
Title: Corporate QA Manager	Date: 5/1/98

TOTAL ORGANIC CARBON-SHIMADZU TOC ANALYZER

1.SCOPE AND APPLICATION

1.1 This method is applicable to the determination of total organic carbon (TOC) in ground water, surface water, drinking water, domestic and industrial wastewater, soils, sediments, and other solids.

1.2 The linear range for TOC aqueous samples is from 1mg/L to 500mg/L for a 250uL injection volume. Samples with TOC concentrations greater than 500mg/L require dilution before analysis. The linear range for soils and solids samples is from 500mg/kg to 235000mg/kg for a 100mg sample.

1.3 The reporting limit(RL), the method detection limit(MDL), and the accuracy and precision criteria are given in Table 5 of the Savannah Labs Comprehensive Quality Assurance Plan (1) or client-specified QAPP.

2.SUMMARY OF METHOD

2.1 Total organic carbon (TOC) is measured by treating a sample with hydrochloric acid (HCl) and purging the sample to eliminate the inorganic carbon present in the sample. The HCl converts any carbonates present in the sample to CO2, which is then purged out of the sample. Purging the sample also eliminates any volatile organic compounds from the sample matrix; therefore, any direct measurement of TOC is actually non-purgeable TOC. The acid treated sample is then combusted at 680C for liquids and 900C for soils. The carbon is converted to carbon dioxide and the carbon dioxide is measured by an infrared (IR) detector. The amount of carbon dioxide, expressed as carbon, is directly proportional to the carbon content of the sample.

2.2 It is important that the analyst know the type of carbon analysis to be determined so that the proper sample can be used for the analysis. The following "equation" shows the relationship between the various forms of carbon that can be measured with this procedure:

$$TC = TOC + TIC$$

where

TC = total carbon
TOC = total organic carbon
TIC = total inorganic carbon

Since TOC has two components, purgeable and non-purgeable, the "equation"can be expanded:

$$TC = npTOC + pTOC + TIC$$

where npTOC = non-purgeable TOC
pTOC = purgeable TOC

SL SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC.

Three of these forms of carbon can be measured directly- TC, npTOC, and TIC. The pTOC has to be determined by difference.

In most cases, the pTOC is very small compared to the npTOC and the npTOC is reported as the "total organic carbon".

2.3 This procedure is based on EPA Method 415.1(2), SW-846 Method 9060(3), and the SL COMPQAP(1).

3.SAFETY

3.1 The analyst must protect herself/himself from exposure to the sample matrix. Many of the samples analyzed for carbon content are potentially hazardous. The analyst must wear protective clothing (lab coat or apron), eye protection (eyeglasses or face shield), and disposable gloves when handling the samples.

3.2 The furnace on the carbon analyzer and on the soil analyzer must be cooled to room temperature before maintainence can be performed. The temperature of the analyzer furnace can reach 680C and the soil furnace can reach 900C. The analyst must be very careful to avoid touching these very hot surfaces.

3.3 Care must be taken when handling hydrochloric acid(HCl) and solutions of hydrochloric acid. The inorganic carbon reagent is 2N HCl and must be handled with caution. This acid will cause chemical burns and destroy unprotected clothing. The analyst must also use extreme caution when adding acid to soil samples. The acid should be added slowly to minimize potentially violent reactions that will splatter acid.

3.4 The carrier gas MUST remain on while the instrument is in operation. The analyst must ensure that there is sufficient carrier gas available in the tank to complete the run. Failure to have carrier gas running through the instrument while the combustion furnace is at the operating temperature may result in melting of the combustion tubes.

3.5 The Material Safety Data Sheets(MSDS) for each regent and standard used in this procedure are available for inspection and review by each analyst at each lab division. The MSDS contain information on the potential hazard that each reagent poses and the guidelines for safely handling the reagent. The MSDS also contains some first aid information and should be taken to the first aid center or hospital with any person that requires a higher level of medical attention after exposer to the reagent.

4.INTERFERENCES

4.1 Sample particulates that are larger than the syringe will not be included in the carbon determination. This condition may result in low results if the particulates contain measurable levels of carbon. The analyst should be alert to samples that may clog the sample syringe.

4.2 Liquid samples containing oil or are oily in nature will deactivate the catalyst. The catalyst will have to be regenerated or replaced prior to running other samples. It is highly recommended that oily

samples be analyzed using the soil module or that the CCV and CCB be analyzed immediately after these samples to evaluate the condition of the catalyst.

4.3 It is very important that the inorganic carbon be removed from the sample prior to the direct determination of the TOC (non-purgeable TOC). Failure to remove the inorganic component of the sample may result in artificially high TOC results.

5.SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Aqueous samples for total organic carbon are collected in 125-mL amber glass containers with zero headspace and preserved with HCl to pH < 2. A light blue dot is placed on the container to indicate that the sample has been preserved with HCl. The sample is iced at the time of collection and is stored at 4C +/- 2C until the time of analysis.

The pH of each aqueous sample for TOC must be determined and recorded when the sample arrives in the lab to ensure proper preservation.

5.1.1 Pour a small aliquot of the well-mixed sample into a small plastic cup.

5.1.2 Dip a piece of wide range pH paper into the sample in the cup. NEVER dip the pH paper into the sample container.

5.1.3 If the pH is less than 2 (<2), the preservative is correct and the sample can be stored in the cooler.

5.1.4 If the pH of the sample is greater than 2 (>2), adjust the pH to less than 2 (<2) with small aliquots of 1:1 HCl solution. Thoroughly mix the acid and sample and re-test another aliquot of the sample. Continue adding small aliquots of acid to the sample until the pH is less than 2. Record the pH of the sample along with any adjustments and/or comments on the TOC sample pH log.

5.2 Soil and sediment samples are collected in 125-ml amber glass containers with minimum headspace. The samples are iced at the time of collection and are maintained at 4C +/- 2C until the time of analysis.

5.3 The hold time for an aqueous sample preserved with HCL is 28 days from the date of collection. The maximum hold time for soil and sediment samples is 28 days from the date of collection.

6.APPARATUS AND MATERIALS

6.1 Shimadzu 5050A Carbon Analyzer

6.2 Volumetric pipets

6.3 Filters (0.45um) and disposable filters

6.4 Volumetric flasks

6.5 Disposable transfer pipets

6.6 Ceramic boats for soil analyses

6.7 Aluminium pans

6.8 Ultra pure compressed air, regulator and appropriate fittings. The compressed air must contain less than 1ppm of carbon dioxide. This gas is used as the carrier and sparge gas for the analysis of aqueous samples.

6.9 Oxygen, regulator, and appropriate fittings. The oxygen is used as the carrier gas in the analysis of soil samples.

7. REAGENTS

7.1 Reagent water- lab-generated deionized water

7.2 Hydrochloric acid (HCl)-concentrated, reagent grade or better. Record the acid lot number in the reagent prep log.

7.3 1:1 Hydrochloric acid solution (50% HCl)- Add approximately 400mL of reagent water to a 2.0-L beaker. Place the beaker on a magnetic stir plate located under a hood. Add a Teflon stir bar to the reagent water and turn the stir plate on. SLOWLY and CAREFULLY add 500mL of concentrated HCl to the beaker in small aliquots. Hydrochloric acid is extremely toxic and has a suffocating odor. After the entire 500-mL of HCl has been added, continue mixing and allow the solution to cool. After the solution has cooled, transfer it to a 1.0-L volumetric flask and dilute to volume with reagent water. This solution should be stored in a glass container. Prepare this solution every six months or as needed. Record the preparation in the reagent prep log.

7.4 2 N HCl Solution-Slowly add 42mL of concentrated HCl to about 150mL of reagent water contained in a 250-mL volumetric flask. Dilute to volume with reagent water. Transfer the reagent to a glass container. This reagent must be prepared under a hood. HCl has a suffocating odor and can cause severe chemical burns.

7.5 Vanadium pentoxide-reagent grade

7.6 Tungsten oxide-reagent grade

8.STANDARDS

8.1 Stock standards

8.1.1 Potassium hydrogen phthalate (KHP)-reagent grade or better.

8.1.2 Potassium hydrogen phthalate, 2000mg Carbon / liter
Dissolve 0.4256g of reagent grade KHP in approximately 800mL of reagent water contained in a 1.0-L volumetric flask. Dilute to volume with reagent water and transfer the stock standard to an amber glass container. Label the container with the standard identification, concentration, analyst's initials, date prepared, and the expiration date. Record the preparation in the standards prep log. Store the standard in the refrigerator

at 4C +/-2C. Prepare this standard monthly or as needed.

8.2 Calibration Standards-TOC

The KHP stock standard is used to prepare the calibration standards for the determination of TOC. The pH of each calibration standard is adjusted to less than 2 with 1:1 HCl. The following table gives the volumes of stock standard needed to prepare the given volume of the calibration standard:

Calibration Standard (mg/L)	mL of 2000mg/L Stock Standard	mL of Reagent Water	Final Volume
500	250	750	1000
200	100	900	1000
100	50	950	1000
50	25	975	1000
10	5.0	995	1000
5.0	2.5	997.5	1000
1.0	0.50	999.5	1000

The concentration of the calibration standard can be determined using the following equation:

$$(Cs)(Vs) = (Ccal)(Vcal)$$

$$Ccal = \frac{(Cs)(Vs)}{(Vcal)}$$

where

Cs = concentration of the stock standard (mg/L)
Vs = volume of stock standard needed to prepare the calibration standard(L)
Ccal = concentration of the calibration standard to be prepared (mg/L)
Vcal = volume of the calibration standard to prepare (L)

9. SAMPLE PREPARATION

9.1 There is essentially no sample preparation for aqueous samples. The samples are loaded on to the autosampler and the sampling sequence is initiated. The analyst should be alert for samples with high solids and particulates that may clog the syringe or may produce results that are lower than might expected.

NOTE: It may be difficult to establish correlation between the TOC, BOD, and COD for samples with high particulates. The syringe system used by the Shimadzu instrument may effectively filter out particulates that may be included in the determination of BOD and COD.

9.2 Soil samples for TOC analysis are dried to produce a free flowing, homogeneous sample. The dried sample is treated with concentrated HCl to convert the carbonates and bicarbonates to carbon dioxide, which evolves from the sample.

9.2.1 Weigh approximately 5g to 10g of the soil or sediment sample into an aluminum pan.

9.2.2 Place the pan in the oven and allow the moisture to evaporate over night at 105C.

NOTE: Heating the soil will drive off the volatile organics. The TOC measured from the soil is the non-purgeable or non-volatile component of the total organic carbon. Since the volatiles are usually very low compared to the non-volatile, the non-volatile component is reported as the TOC.

9.2.3 Remove the samples from the oven. Crush any lumps that may be present with a spatula or spoon and mix the sample to obtain a homogeneous sample.

9.2.4 Weigh out five (5) 100mg portions of the sample into the ceramic "boats". Record the weight of each sample to the nearest 0.1mg on the analysis log.

9.2.5 Add approximately 0.10mL of concentrated HCL to each ceramic "boat". Additional HCl can be added if the soil has a high carbonate content. This step should be performed in a hood.

9.2.6 Place the sample "boats" into the oven to allow the excess HCL to evolve. The sample is now ready for analysis.

10.PROCEDURE

10.1 Aqueous Samples-TOC

10.1.1 Turn the Shimadzu 5050A on and set the following parameters:

Analyzer Furnace: 680 C
Carrier Gas: 150mL/min (ultrapure compressed air)
Purge Gas: 50mL/min (ultrapure compressed air)
Injection Volume: 250uL (0.250mL)
Minimum Number of Replicates: 3
Maximum Number of Replicates: 5

Allow the instrument to equilibrate and the furnace to reach the set point before processing samples.

10.1.2 Set up the calibration standards on the autosampler. The lowest standards should be analyzed first. The analytical sequence is :

Range 1 Calibration Standards:
1.0mg/L
5.0mg/L
10mg/L

Range 2 Calibration Standards
1.0mg/L
10mg/L
50mg/L
100mg/L

Range 3 calibration Standards
1.0mg/L
100mg/L

200mg/L
500mg/L

Initial Calibration Verification(ICV/LCS)
Initial Calibration Blank (ICB)
10 client samples (in replicate)
Continuing Calibration Verification(CCV/LCSD)
Continuing Calibration Blank (CCB)
10 client samples (in replicate, including an MS/MSD per batch of twenty samples)
CCV/LCS
CCB

This sequence continues until there are no more samples to fill the slots. The CCV and CCB must "cap" the end of the analysis sequence. Note that the ICV and the CCV are from a separate source (ERA) and are evaluated for calibration verification and overall analytical control(accuracy and precision)

The initial calibration is performed each time samples are analyzed.

10.1.3 Record the sample identifications on the analysis log sheet to correspond to the position occupied by the samples on the autosampler tray.

10.1.4 Start the analyzer. The samples will be processed automatically.

10.1.5 After the sequence has ended, evaluate the QC and the sample results.

10.1.5.1 If the ICV or a CCV or the ICB or a CCB has failed, the samples analyzed between the failed CCV or CCB must be re-analyzed. The result for the ICV/CCV must be 80-120% of the true value. The results for the ICB/CCB must be less than the quantitation limit of 1mg/L.

10.1.5.2 If a sample contains a concentration of TOC greater than the linear range of the calibration curve, the sample must be diluted and re-analyzed.

10.2 Soil Samples-TOC The TOC analyzer is initially calibrated using a 1-point calibration curve on each analysis range. The curve is plotted from zero through the area corresponding to the standard for the specified range. The calibration levels are:

RANGE	mg KHP	mg Carbon
X1	2.1	1.0
X5	8.0	3.8
X30	50	23.5

These standards define the linear range of the instrument.

10.2.1 Turn the Shimadzu 5050A and soil analyzer module on and set the following parameters:

Soil Analyzer Furnace: 900 C
Carrier Gas: 50mL/min (oxygen)

11.2.1.1 The theoretical concentration of an aqueous sample that has been spiked with the matrix spiking solution is

$$mg/L = \frac{0.5mL \times 2000mg/L}{100mL} = \frac{0.0005L \times 2000mg/L}{0.100\ L} = 10mg/L$$

11.2.1.2 The theoretical concentration of a soil sample that has been spiked with a 20mg portions of dextrose is:

$$mg/kg,dw = \frac{20mg \times 0.40mg\ C/mg}{100mg} = \frac{8mg\ C}{100mg} = 80000\ mg/kg$$

11.2.1.3 The percent recovery of the matrix spike in the matrix spike sample is calculated:

$$\%recovery\ (\%REC) = \frac{(Cms - Cs)}{Ts} \times 100$$

where

Cms = concentration of the spiked sample (MS or MSD)
Cs = concentration of the unspiked sample
Ts = theoretical concentration of the spike

11.2.1.4 The matrix spike samples are analyzed in duplicate. The relative percent recovery(%RPD), a measure of precision, is calculated for the MS/MSD pair as follows

$$\%RPD = \left| \frac{\%REC(MS) - \%REC(MSD)}{(\%REC(MS) + \%REC(MSD))/2} \right| \times 100$$

11.2.2 The lab control standard is purchased from ERA (Environmental Resource Associates). The standard comes in a vial that is diluted to a specified volume with reagent water. The results are compared to the values contained on the certificate of analysis. The lab control sample for soil samples is dextrose. Twenty milligrams of dextrose is weoghed into a ceramic boat and analyzed as a sample. An EPA sample, municipal digested sludge, is also analyzed as a control standard.

11.2.2.1 The recovery of the lab control standard is calculated as follows:

$$\%REC = \frac{Clcs}{Ct} \times 100$$

Where

Clcs = concentration of the LCS or LCSD
Ct = theoretical concentration of the LCS or LCSD

11.2.2.2 The lab control standards samples are analyzed in duplicate. The relative percent recovery(%RPD), a measure of precision, is calculated for the LCS/LCSD pair as follows

Allow the instrument to equilibrate and the furnace to reach the set point before processing samples.

10.2.2 Before samples are processed, a 1.0-mL aliquot of 2N HCl is combusted in the furnace to clean and regenerate the catalyst.

10.2.2.1 Add 0.10mL of 2N HCl to the ceramic "boat"

10.2.2.2 Add sufficient ceramic "wool" to cover the acid.

10.2.2.3 Place the "boat" into the furnace and allow the sample to cycle through the combustion and cooling steps.

10.2.3 A method blank, consisting of the ceramic "boat", ceramic "wool" and metal catalyst (vanadium pentoxide or tungsten oxide), is then processed through the combustion and cooling cycle to evaluate the contribution of the "boat", ceramic "wool", and the metal catalyst to the overall TOC analysis. If the weight of a "blank sample" is assumed to be 100mg, the method blank should be less than 500mg/kg,dw.

10.2.4 After the method blank has been analyzed, the following QC Check Standards are analyzed: four 20-mg portions of dextrose and one 15-mg to 18-mg portion of the EPA Municipal Digested Sludge. The recovery of each of these check standards must be within the SL COMPQAP Table 5 criteria for recovery. The duplicate analyses of the dextrose standards ar e used to evaluate precision, which must be less than the Table 5 limit for %RPD

10.2.5 The client samples are prepared as described in Section 9.2. It is important that all of the inorganic carbon be converted to CO2 prior to the analysis of the soil for TOC.

10.2.6 One of the five prepared portions of soil is analyzed on the X5 range to determine the proper range for that particular sample.

10.2.6.1 If the result for the sample is within the X5 range, that is , if the result is quantifiable but less than the high standard (within the linear range), the X5 range is used and three of the other portions are analyzed on the X5 range. Each sample must be analyzed in quadruplicate.

10.2.6.2 If a peak is not quantified, a second 100mg portion of the sample is analyzed on the X1 range. The three remaining portions of sample are analyzed on the X1 range. Each sample must be analyzed in quadruplicate.

10.2.6.3 If the concentration of the initial analysis exceeds the linear range on the X5 range, a second portion of the sample is analyzed on the X30 range. If the result on the X30 range is within the linear range, the remaining portions are analyzed on the X30 scale. Each sample must be analyzed in quadruplicate.

If result is above the linear range on the X30 range, a smaller aliquot of sample should be weighed and analyzed.

10.2.7 Each soils sample must be analyzed in quadruplicate on the same range setting. The average result of the four replicates is reported unless a request is made for the result of each individual replicate.

11. DATA ANALYSIS/CALCULATIONS

11.1 The concentrations of TOC in client and QC samples are taken from the linear regression curve.

11.1.1 The results for liquid samples are calculated as follows:

mg/L (in sample) = mg/L (from printout) x DF

where
DF = dilution factor

11.1.2 The Reporting Limit (RL) is calculated:

$RL(mg/L) = RL_{qap} \times DF$

where RL_{qap} = reporting limit from Table 5, SL COMPQAP
DF = dilution factor

The SL Quality Assurance Plan Reporting Limit assumes that DF=1.

11.1.3 The results for soil sample is calculated as follows:

mg/kg,dw = mg/kg(from curve)

Since the sample is dried prior to analysis, the result from the printout is given on a "dry weight basis". The weight of the soil is entered into the data system and the result is calculated as mg/kg, dw since the sample is dried prior to analysis.

11.1.4 The Reporting Limit (RL) is calculated:

$$mg/kg,dw = RL_{qap} \times \frac{100mg}{W}$$

where
RL_{qap} = reporting limit from Table 5, SL COMPQAP
W = weight of sample analyzed (mg)

The SL Quality Assurance Plan Reporting Limit and assumes that 100mg(0.100g) of sample is analyzed.

11.2 The concentration of TOC 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.

11.2.1 0.0mL of the 50mg/L matrix spiking solution is added to a 250mL aliquot of a liquid sample or to 10.0g of a soil sample(the soil sample is mixed with 250mL of reagent water)

$$\%RPD = \left| \frac{\%REC(LCS) - \%REC(LSCD)}{(\%REC(LCS) + \%REC(LCSD)) / 2} \right| \times 100$$

12. QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items. The QC items for an analytical batch consist of a method blank, a lab control standard(LCS), a lab control standard duplicate(LCSD), a matrix spike(MS), and a matrix spike duplicate(if there is sufficient volume to perform the MS/MSD)

12.2 **Evaluation of QC Data**

12.2.1 The **method blank** is reagent water that is taken through all of the analytical procedures. The concentration of TOC in the method blank is a measure of the contamination due to the entire analytical process.

12.2.1.1 The concentration of TOC in the method blank must be less than 1.0mg/L for liquids and 500mg/kg for soils(Assuming a "soil" weight of 100mg(0.1000g).

12.2.1.2 If the concentration of TOC in the method blank is greater than 1.0mg/L for liquids and 500mg/kg for soils, contact the immediate supervisor to determine the cause of the contamination. If the cause of the contamination cannot be determined and corrected, the batch of samples processed with the method blank must be reanalyzed.

12.2.2 The **lab control standard (LCS or LCSD)** is a standard obtained or prepared from a separate source (a source different from the source of the calibration standards) and taken through all of the analytical procedures. The LCS for TOC is purchased from ERA. A certificate of analysis must accompany the lab control standard and the certificate must be kept as a record of the true (theoretical) concentration of the LCS.

12.2.2.1 The recoveries(%REC) of the LCS and LCSD must be between 80% and 120%. The relative percent difference(%RPD) between the LCS and LCSD must be less than 20%

12.2.2.2 If the recovery of TOC in the LCS or LCSD is outside of the 80-120% range, contact the immediate supervisor to determine the cause. If the %RPD of the LCS/LCSD is greater than 20%, contact the immediate supervisor to determine the cause. If the cause of the failure of either the %REC or %RPD cannot be determined and corrected, the batch of samples processed with the LCS/LCSD must be reanalyzed.

12.2.3 The **matrix spike and matrix spike duplicate (MS/MSD)** is prepared by adding a known concentration of a TOC standard to a known volume or weight of a client sample. The spiked samples are processed through the entire analytical procedure. The accuracy and precision results from the MS/MSD are a measure of the effect that the sample matrix has on the determination of TOC.

12.2.3.1 The recoveries(%REC) of the MS and MSD should be between 60% and 140%. The relative percent difference(%RPD) between the MS and MSD should be less than 40%.

12.2.2.2 If the recovery of TOC in the MS or MSD is outside of the 60-140% range, contact the immediate supervisor to determine the cause. If the %RPD of the LCS/LCSD is greater than 40%, contact the immediate supervisor to determine the cause. If the cause of the failure of either the %REC or %RPD cannot be determined and corrected and the LCS/LCSD are within the specified control limits, the cause can be attributed to matrix interference(s).

NOTE : FOR PROJECTS WHICH REQUIRE FLORIDA DEP QAS CRITERIA, MATRIX SPIKE RESULTS WILL BE UTILIZED FOR LABORATORY CONTROL. IF THE MATRIX SPIKES ARE OUT OF CONTROL, LABORATORY CONTROL STANDARD (LCS) AND METHOD CONTROL CRITERIA WILL BE UTILIZED FOR ULTIMATE CONTROL OF THE ANALYTICAL BATCH.

12.3 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. The MDL is determined by spiking 10 aliquots of reagent water or 10 aliquots of blank soil (a soil that has been analyzed previously and found to contain no TOC above the RL) with a known concentration of TOC. The spiked samples are taken through the analytical process. The results of the analysis of the spiked samples is used to calculate the MDL.

12.3.1 Prepare the spiked samples for the determination of the Method Detection Limit.

12.3.1.1 Spike ten(10) 100-mL aliquots of reagent water with 0.10mL of the TOC matrix spiking solution (2000mg/L) to determine the MDL for liquid samples. The concentration of TOC in the water sample is 1.0 mg/L.

12.3.1.2 Spike ten(10) aliquots of a blank soil (a soil that has been analyzed previously and found to contain no TOC above the RL) with 0.025mL of the TOC matrix spiking solution (2000mg/L) to determine the MDL for soil/solid samples. The concentration of the TOC in the soil sample is 1000 mg/kg.

12.3.2 Analyze (Section 10.) the MDL samples. Calculate the concentration of the samples (Section 11.). Enter the data on the LIMS sheets. A LIMS-based program will calculate the MDL. A report will also be generated and returned to each section as a record of the MDL

13. REFERENCES

(1) Savannah Laboratories *Comprehensive Quality Assurance Plan*, 1998

(2) *Method for Chemical Analysis of Water and Wastes;* U.S. EPA Office of Research and Development: Cincinnati, OHIO, March, 1983

(3) *Test Methods for the Evaluating Solids Wastes, Third Edition, SW-846*; US EPA Office of Solid Waste and Emergency Response: Washington, DC.

Approval Signature:	R. Wayne Robbins
Title: Corporate QA Manager	Date: 5/1/98

TOTAL ORGANIC CARBON: DOHRMANN DC-80 METHOD

1.0 SCOPE AND APPLICATION

This method is used to determine total organic carbon (TOC) in drinking water, groundwater, and wastewater, as well as some sludges. In addition, procedures are given for determining total carbon (TC), total inorganic carbon (TIC), and dissolved organic carbon (DOC).

2.0 SUMMARY OF METHOD

Organic carbon in liquid samples is determined by oxidation with a persulfate solution in a digester cell, accelerated with UV light. The organic carbon present in the sample is oxidized to CO_2 which is swept by an O_2 stream to an infrared detector. Samples are acidified and purged prior to instrumental analysis to remove inorganic carbon in the form of carbonates and bicarbonates.

Total carbon in liquid samples can be determined by not acidifying the samples upon collection and omitting the purging step prior to analysis.

Total inorganic carbon can be determined in liquid samples by not acidifying and not purging the samples prior to analysis. In addition, the persulfate digestion reagent is replaced by a 10% H_2SO_4 solution and the UV light is turned off.

Dissolved organic carbon can be determined by filtering the sample through a 0.45-µm membrane filter prior to acidification. The sample is then analyzed as it would be for total organic carbon.

This standard operating procedure is based on EPA Method 415.1(1), Standard Method 5310C(2), and SW-846 method 9060(3).

3.0 INTERFERENCES

In TOC analysis, inorganic carbon is removed from the samples by acidification and purging with oxygen. This may remove easily volatilized substances containing organic carbon, however. If the sample is suspected of containing significant quantities of organic carbon that will be purged by this procedure, the total carbon and the total inorganic carbon can be determined in the sample. The difference of the two values should be the true total organic carbon, including the purgeable organic carbon.

Samples containing substances composed of difficult-to-oxidize organic carbon may be slowly oxidized and result in "timeout" errors. This problem can be resolved by injecting a smaller amount of sample, or dilution and reanalysis of the sample.

Relatively high concentrations of chloride in the sample can interfere in the oxidation process. This can result in timeout errors. If samples are suspected of containing high chloride concentrations, a mercury-containing persulfate solution is substituted for the normal digestion reagent.

Contamination can be a problem in total organic carbon determinations. Minimize contamination by careful handling of samples, standards, and blanks, and insuring the use of clean labware.

4.0 APPARATUS AND MATERIALS

Dohrmann DC-80 TOC analyzer, or equivalent instrument
Furnace attachment for DC-80
Syringes, 250- and 1000-µL
Medicine cups
Volumetric glassware

5.0 REAGENTS AND STANDARDS

Digestion reagent, 2% potassium sulfate: Dissolve 20 g of reagent grade potassium persulfate ($K_2S_2O_8$) in some DI water. Add 1 mL of concentrated phosphoric acid (H_3PO_4), and dilute to 1 L. Store in a cool dark place.

Mercuric digestion reagent: Dissolve 8.2 g reagent grade mercuric chloride ($HgCl_2$) and 9.6 g reagent grade mercuric nitrate monohydrate ($Hg(NO_3)_2 \cdot H_2O$) in 400 mL of DI water. Add 20 g of reagent grade potassium persulfate ($K_2S_2O_8$) and 5 mL concentrated nitric acid (HNO_3). Mix well and dilute to 1 L with DI water.

TOC stock standard, 2000-mg/L: Dissolve 0.425 g (dried 1 h at 105° C) reagent grade potassium hydrogen phthalate (KHP) in some DI water and dilute to 100 mL. Standards are prepared fresh at least monthly.

TOC working standard, 400-mg/L: Pipet 20 mL of 2000-mg/L standard into a 100-mL volumetric flask. Dilute to volume with DI water.

TOC working standard, 10-mg/L: Pipet 5 mL of 2000-mg/L standard into a 1-L volumetric flask. Dilute to volume with DI water.

Matrix spikes: Spike samples at 100 mg/L using stock standard solution.

Oxygen gas, commercial grade

TIC cell solution: Dilute 0.3 mL concentrated sulfuric acid (H_2SO_4) to 1 L with DI water.

TIC calibration standard, 400-mg/L: Dissolve 3.532 g of dry reagent grade sodium carbonate (Na_2CO_3) in some DI water. Dilute to 1 L.

TIC independent check standard, 40-mg/L: A 400-mg/L stock check standard is prepared as above, but independently from standard used for instrument calibration. A volume of 50 mL of the 400-mg/L stock is then diluted to 500 mL with DI water to yield the 40-mg/L independent check standard.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Samples for TOC analysis are collected in 125-mL amber glass bottles with no headspace, preserved with H_2SO_4 to a pH < 2 (green dot), and cooled to 4° C. Holding time for TOC samples is 28 days.

Samples for TC and TIC analyses are collected as above, but no preservative is added to the sample (purple dot). Samples are refrigerated, however.

Samples for DOC analysis are collected as above, with no preservative added to the sample bottles. Samples are filtered with pre-cleaned 0.45-µm filters, as soon as possible after collection.

7.0 PROCEDURE

Turn on the oxygen to the instrument. Turn on the DC-80 by depressing the "power" button. Allow the instrument to warm up for 20 min. The oxygen flow rate should be 200 mL/min (regulator should be set at 35 psi).

For routine operation, "200 uL" is selected as the injection volume.

Select the "det" function to check baseline stability. This allows one to read to real-time CO_2 concentration in the detector. The readings on the digital readout should be reasonably stable. If not, this indicates a possible oxygen leak, which should be corrected prior to calibration and analysis.

In addition to being stable, the reading of the baseline should be between 0.0100 and 0.0110. If it is not within this range, set it by adjusting the "zero" adjustment with a screwdriver. The instrument is now ready for calibration. The instrument should be calibrated daily or upon instrument startup. Load 200 uL of the 400-mg/L standard into the syringe. Press the "start" button and inject the standard. After the instrument has completed integration of the sample (the instrument beeps and the "ready" light comes on), the "calibrate" button is pressed momentarily (< 1 s). This averages and stores the calibration result.

Up to seven calibration results can be averaged and stored by the instrument.

If the calibration result appears lower than normal, all old standard values can be erased by pressing the calibrate button until the light goes off.

After calibration, an independent calibration verification standard is analyzed, followed by a blank. If the calibration verification standard and blank meet control criteria, these are followed by the analysis of 10 samples.

Prior to the analysis of samples, they must be purged to rid them of any CO_2 from inorganic carbon remaining in solution. A sample is purged by pouring a portion of it into a medicine cup, placing the metal probe into the cup, turning on the oxygen with the toggle switch on the side of the instrument, and allowing it to sparge for 5-6 min.

After purging, the samples are injected into the instrument and analyzed. Sample TOC concentration is read from the LED readout on the instrument when the function switch is set to "ppm." Sample TOC concentrations are also printed out on a paper tape.

After 10 samples are analyzed, another QC-check standard is analyzed, followed by another blank. All QC data bracketing the 10 samples must meet acceptance criteria in order for the sample data to be acceptable.

Samples which contain high concentrations of TOC or difficult-to-oxidize organic matter (indicated by the "error" light coming on) may require dilution or use of the furnace technique. In order to get a representative sample, samples containing suspended matter should not be too highly diluted. For these types of samples and sludges, the furnace technique should be used.

Samples containing high concentrations of chloride may also result in "timeout" errors. If samples are suspected of this problem, remove the digestion reagent from the digestion cell and receptacle and replace with the mercury containing reagent.

8.0 QUALITY CONTROL/QUALITY ASSURANCE

A single-point calibration is performed daily or upon instrument power-up with a 400-mg/L TOC

standard. Occasionally, for groups of high level samples, calibration is performed with a 2000-mg/L TOC standard.

A calibration verification standard is analyzed upon calibration, after every 10 samples, and at the end of every run.

A blank is analyzed immediately after each calibration verification standard.

All QC data bracketing the 10 samples must meet acceptance criteria in order for the sample data to be acceptable.

A matrix spike and a matrix spike duplicate are analyzed for each batch or each 20 samples, whichever is more frequent.

If SW-846 method 9060 for TOC is specified, samples are analyzed in quadruplicate.

Samples with TOC concentrations < 10 mg/L are analyzed in duplicate.

A linearity check is performed monthly, by the analysis of 5-, 25-, 100-, 200-, and 400-mg/L standards. The correlation coefficient for the linear least squares line through the points should be > 0.995.

For TC determinations, QC is as above. For TIC, QC as for TOC and TC, except substituting TIC standards for TOC standards.

For DOC determinations, QC is as given above for TOC. In addition, if samples are not filtered in the field, two filtered blanks and two filtered standards are analyzed for every batch or every 20 samples.

9.0 REFERENCES

1. *Methods for Chemical Analysis of Water and Wastes*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983.

2. *Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition*; American Public Health Association: Washington, DC, 1992.

3. *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

4. *Dohrmann DC-80 Total Organic Carbon Systems Manual*; Santa Clara, CA, July, 1986.

TOC WORKSHEET

EPA Method 415.1

Date	Initials	Sample Log #	Sample ID	Volume Used (uL)	Vial #	Tape #	TOC	TOC Avg.	Comments

BA51:08.26.97:0

Approval Signature: R. Wayne Robbins

R. Wayne Robbins

Title: Corporate QA Manager Date: 8/29/97

PERCENT SOLIDS/PERCENT MOISTURE FOR REPORTING SOILS ON A "DRY-WEIGHT BASIS'

1.0 SCOPE AND APPLICATION

This method is applicable to the determination of the percent solids (or percent moisture) in soils, sediments, sludges, and other solid materials that must be reported on a "dry weight basis".

2.0 SUMMARY OF METHOD

2.1 A well mixed sample is transferred to a tared aluminum weighing pan or crucible and the sample is placed in an oven maintained at 103C-105C. The residue that remains after the liquid has been evaporated is the solids portion of the sample. The solids can be expressed either as the percent solids or as the percent moisture.

2.2 This procedure is based on the percent solids determination in SW-846 Methods 3050 and 3550.

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 must protect himself/herself from exposure to the sample matrix. Many of the samples that tested for percent solids may contain hazardous chemical compounds or biological organisms . The analyst must wear protective clothing (lab coat or apron), eye protection (glasses or face shield), and disposable gloves when handling these samples.

3.3 The analyst should handle samples that have been dried at 103C - 105C with caution. This temperature can cause skin burns.

3.4 The MSDS for the reagents used in this procedure are on file in the laboratory and are available for reference and inspection.

4.0 INTERFERENCES

The major cause of interferences will be from glassware or other containers that have not been properly cleaned or prepared prior to the analysis. Since the basis of this procedure is the difference in the weight of the aluminum pan or crucible containing the residue and the tare weight of the crucible, care must be taken to ensure that the aluminum pan or crucible is not treated in such a manner to add or lose weight.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

Aliquots of samples for the determination of percent solids are routinely sub-sampled from the 100mL to 500-mL widemouth containers collected for metals or organic extractable analyses. These samples are iced at the time of collection and are maintained at 4C+/-2C until the time of analysis. The percent solids should be determined as soon as possible.

6.0 APPARATUS AND MATERIALS

6.1 Aluminum pans

6.2 Top-loading balance-capable of weighing to the nearest 0.01g

6.3 Drying oven-capable of maintaining a temperature of 103-105C

6.4 Spatula or other utensil for transferring sample

7.0 REAGENTS

No reagents are required for this procedure.

8.0 STANDARDS

Calibration and check standards are not required for this analysis.

9.0 SAMPLE PREPARATION

Sample preparation steps are included in Section 10.

10.0 PROCEDURE

10.1 Calibrate and verify that the top loading balance is working within the proper parameters in accordance with SL SOP AN10:*Balance Calibration and Use.*

10.2 Label an aluminum pan with an identification number. Weigh the pan on the top loading balance. Record the ID and the weight (to the nearest 0.01g) of the aluminum pan on the bench sheet.

10.3 Tare the balance by pressing the auto tare button. This will zero the balance.

10.4 Thoroughly mix the sample with a stainless steel spatula or glass rod. It is important to obtain a homogeneous mixture prior to sub-sampling so that the sub-sample will accurately reflect the composition of the sample. Leaves, rocks, and other foreign materials should not be included in the sub-sample.

NOTE: If there is any doubt as to how to treat a given sample, contact the immediate supervisor to determine the proper course of action. SL SOP AN70 provides guidance for homogenizing samples.

10.5 Add a 5g to 10 g aliquot of the well-mixed sample to the tared aluminum pan. Record the sample identification and weight of the sample to the nearest 0.01g on the analysis log.

10.6 Place it in the drying oven maintained at 103-105C. The sample should remain in the oven overnight.

10.7 Remove the aluminum from the oven, allow them to cool slightly, and place them in the desiccator. Remember not to place the aluminum pan on a surface that can cause dirt or other foreign objects to adhere to the pans. After the pans are cool, place them in the desiccator.

10.8 Allow the aluminum pans to remain in the desiccator until completely cool.

10.9 Weigh the aluminum pan with the sample residue on the top-loading balance and record the weight of the pan and the residue to the nearest 0.01g. Calculate the total solids (percent solids) using the equation in Section 11.

11.0 CALCULATIONS

11.1 The percent solids is calculated using the following equation:

$$percent\ solids = \frac{A - B}{W} x\ 100$$

where

A = weight of sample residue and aluminum pan (g)
B = weight of aluminum pan (g)
W = weight of sample used to determine the percent solids (g)

To express the percent solids as percent mixture:

$$\text{Percent moistue} = 100 - \text{percent solids}$$

To express the percent solids as a decimal equivalent ("solids") for calculating sample results on a "dry weight basis":

$$\text{Soilds} = \frac{\text{percent solids}}{100}$$

12.0 QUALITY CONTROL/QUALITY ASSURANCE

The balance must be checked in accordance with SL SOP AN10: *Balance Calibration and Use* prior to use.

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLE-SHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solids Waste, Third Edition*, SW-846; USEPA Office of Solids Waste and Emergency response, Washington, D.C.

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager Date: 3/12/97

pH ELECTROMETRIC MEASUREMENT OF WATER, SOILS, AND WASTES

1.0 SCOPE AND APPLICATION

1.1 This procedure can be used to determine the pH of aqueous samples and soil and waste samples. Aqueous samples include monitoring wells, wastewater, and industrial waste such as influents and effluents. Waste samples include solids, sludges, or non-aqueous waste. The possible range pH is 1 to 14.

This SOP also contains procedures for determining pH with pH paper. pH paper can be used to determine if a sample is properly preserved, if a sample has been adjusted to the proper range for extraction, digestion, or leaching, or to approximate the pH before the pH electrode is employed.

1.2 pH is defined by the following equation:

$$pH = -1 (\log [H^+])$$

This shows that pH is a logarithmic function; that is, an increase or decrease of one pH unit represents a ten-fold change in hydrogen ion concentration. The following table demonstrates the relationship between pH and hydrogen ion concentration:

pH	Hydrogen Ion Concentration
1.0	0.10 (1×10^{-1})
2.0	0.010 (1×10^{-2})
3.0	0.0010 (1×10^{-3})
4.0	0.00010 (1×10^{-4})
5.0	0.000010 (1×10^{-5})
6.0	0.0000010 (1×10^{-6})
7.0	0.00000010 (1×10^{-7})
8.0	1×10^{-8}
9.0	1×10^{-9}
10.0	1×10^{-10}
11.0	1×10^{-11}
12.0	1×10^{-12}
13.0	1×10^{-13}
14.0	1×10^{-14}

2.0 SUMMARY OF METHOD

2.1 The pH of an aqueous sample is determined potentiometrically using a combination electrode with temperature compensation. A soil or waste sample is mixed with reagent water. The pH of the solution is measured with the electrode. This procedure may not be used to determine whether a non-aqueous material exhibits the hazardous characteristic of corrosivity (see Chapter 7 of SW-846 Section 7.2).

2.2 The pH meter/electrode is calibrated using a series of certified buffer solutions that bracket the pH of the sample. This procedure for an aqueous sample is based on EPA Method 150.1, SW-846 Method 9040, and SM4500-H+. This procedure for a soil or waste sample is based on SW-846 Method 9045C.

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 will put you or others in a potentially hazardous situation.

3.2 The analyst should wear a lab coat or apron, gloves, and eye protection when handling the pH buffers and samples.

3.3 MSDS (Material Safety Data Sheets) are available for each reagent and standard used in the lab. The analyst should be familiar with the information supplied in the MSDS.

4.0 INTERFERENCES

4.1 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping with a Kimwipe moistened with hexane followed by rinsing with reagent water. Additional cleaning with hydrochloric acid may be required if the response is sluggish after cleaning with hexane. See Standard Methods 4500-H^+ Section 5b.

4.2 Temperature effects are minimized by the use of a meter/electrode confirmation that automatically compensates for temperature. The temperature of the samples should be at or near the same temperature as the buffer solutions.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Aqueous samples for pH measurement should be collected in 100-mL plastic containers. Waste or soil samples for pH measurement are routinely collected in a 100ml glass or plastic container. All containers should be stored at 4°C ± 2°C until analysis. The pH must be determined as soon as possible after collection.

6.0 APPARATUS AND MATERIALS

6.1 pH Meter: Orion Model 501 or equivalent instrument with combination electrode (Orion 91-55 or equivalent) with temperature compensation.

6.2 50ml beakers or other suitable container

6.3 Magnetic stirrer and Teflon-coated stir bars

6.4 Analytical Balance

6.5 pH Paper - wide and narrow range

7.0 REAGENTS

Reagent Water - Lab generated deionized water

8.0 STANDARDS

8.1 Certified pH buffers to bracket pH range of samples. Suggested buffers are 2.0, 4.0, 7.0, 10.0, and 13.0.

8.2 Check Standard: certified solution from ERA or other vendor.

9.0 SAMPLE PREPARATION

9.1 Sample Preparation of Aqueous Samples

There are no preparation steps for the determination of pH in aqueous samples.

9.2 Sample Preparation of Soil Samples

If there is insufficient soil to use 20g, a smaller aliquot can be used if the 1:1 ratio of sample to water is maintained.

9.2.1 To 20g of soil in a 50ml beaker, add 20ml of reagent water. Add a magnetic stir bar to the beaker and place the beaker on the magnetic stir plate. Stir the suspension for 5 minutes. Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices. If the ratio of water to sample is greater than 1:1, include this information on the reported results.

9.2.2 Let the soil suspension stand for about 1 hour to allow most of the suspended material to settle out from the suspension or filter or centrifuge to obtain the aqueous phase for pH measurement.

9.3 Sample Preparation of Waste Materials

9.3.1 To 20g of waste sample in a 50-mL beaker, add 20mL of reagent water, cover, and continuously stir in the suspension for 5 minutes. Additional dilutions are allowed if working with hygroscopic wastes and salts or other problematic matrices. If the ratio of water to sample is greater than 1:1, include this information in the reported results.

9.3.2 Let the waste suspension stand for about 15 minutes to allow most of the suspended waste to settle out from the suspension or filter or centrifuge to obtain aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the analysis again using 20g of waste and 40mL of reagent water. Include this water to sample ratio with the results.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Step 3.3) if it becomes coated with an oily material. (See Standard Methods 4500-H^+, Section 5b. or SW-846 Method 9045C, Section 3.3).

10.0 PROCEDURE

SEQUENCE
Calibrate with 2 buffers
Check pH with third buffer
Analyze check solution
10 sample determinations
Check Solution
10 sample determinations
Check Solution

10.1 Routine Calibration

This procedure describes the calibration for the range of pH 4 to pH 10 since most pH measurements will fall into that range. Make sure that the pH meter has warmed up and that pH is the selected mode of operation. The lot numbers of the pH buffers must be recorded on the pH data sheet.

10.1.1 Place the pH 7.0 buffer in a beaker on the magnetic stir plate and place the pH electrode in the buffer solution. The electrode should be immersed in the buffer but should not come in contact with the Teflon-coated stir bar.

10.1.2 Allow the solution to stir slowly while the measurement is being made. Adjust the "standardize" or "calibrate" knob on the meter until the pH reads 7.0 ± 0.1.

10.1.3 Rinse the pH electrode with reagent water and place the electrode in the pH 4.0 buffer. Allow the solution to slowly stir while the measurement is being made. Adjust the "slope" or "temperature" knob to bring the meter reading to 4.0.

10.1.4 Rinse the electrode with reagent water and place the electrode in the pH 10.0 buffer. Allow the solution to stir slowly while the measurement is being made. The reading should be 10.0 ± 0.1 pH units. If the buffer does not read within the range, repeat the calibration procedure.

NOTE: It is Savannah Labs policy to consider pH measurements in the 4 to 10 range to be bracketed by two calibration buffers when the meter is calibrated by Section 10.1.

10.2 Non-routine Samples

Samples with pH below 4 and above 10 must be measured with the pH meter/electrode calibrated with buffers that bracket the pH. The pH may be estimated using wide range pH paper and the buffers chosen accordingly. The general procedure is the same described in Section 10.1.

10.3 Sample Analysis

10.3.1 Remove the samples from the refrigerator and allow the samples to come to room temperature. The samples and pH buffers used to calibrate the pH meter/electrode must be at or near the same temperature.

10.3.2 Calibrate and verify the pH meter/electrode. The lot numbers of the pH buffers and check standards must be recorded on the data sheet as described in Section 10.1.

10.3.3 Aqueous Samples

Place an aliquot of the well mixed sample into a beaker on the magnetic stir plate. Make sure that the electrode is immersed in the sample but does not contact the Teflon-coated stir bar.

10.3.3.1 Allow the sample to slowly stir while the pH measurement is being made. Record the pH after the reading has stabilized.

10.3.4 Soil and Waste Samples

Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant to establish good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrode into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

10.3.5 Rinse the electrode with reagent water. Analyze all of the samples in the analytical batch according to the sequence listed earlier in this section.

10.3.6 After all measurements have been made, return the electrode to the 7 buffer or electrode storage solution and set the meter to "standby." The meter should be left "on" when not in use.

10.4 Use of pH Paper

pH paper provides a quick and easy way to approximate the pH of a sample. pH paper can be used to determine if a sample has been properly preserved or if the pH of a sample is in the proper range for a preparation step.

10.4.1 pH paper should be checked upon receipt and quarterly to make sure that it is functioning properly.

-Examine the pH paper. If the paper is discolored or looks worn, it may be defective.
-Place a piece of pH paper on a watch glass or other suitable surface and add a few drops of a certified buffer solution onto the paper
-Compare the color of the pH paper to the reference colors. If the colors match, the paper can be used. If not, acquire new paper.

10.4.2 Determination of pH with pH Paper

Dip a glass rod or disposable Pasteur pipet into the sample and touch the rod or pipet to the pH paper. Compare the color to the reference.

NOTE: Do not dip the pH paper into a sample. The pH paper dye can bleed into the sample and affect sample results.

11.0 CALCULATIONS

The measurement of pH requires no calculation since the reading is taken from the meter. The calculation of percent recovery of the check standard and precision of pH is more complex than it first appears because the pH scale is a logarithmic function. The pH must be converted back to a concentration prior to the calculation of accuracy and/or precision since slight differences in the pH represent large differences in concentration.

11.1 Calculation of hydrogen ion concentration from a pH measurement.

$$pH = -1\ (\log[H^+])$$

$$\frac{pH}{-1} = \log[H^+]$$

$$antilog\ (\frac{pH}{-1}) = [H^+]$$

A calculator that can perform log functions is very handy to make this calculation:

1) enter the pH and change the value to a negative.
2) Take the antilog of the number. (For most calculators that will mean using the 10^x key).

11.2 The percent recovery is calculated:

$$\%Rec = \frac{H}{H_T} \times 100$$

where

H = hydrogen ion from pH measurement
H_T = theoretical hydrogen ion concentration

EXAMPLE

A check standard is analyzed and the pH is determined to be 8.90. The certified concentration is 9.05. The percent recovery is calculated:

$$H_{8.90} = antilog(-8\ .90) = 1.26 \times 10^{-9}$$
$$H_{9.05} = antilog(-9\ .05) = 8.91 \times 10^{-10}$$
$$\%Rec = \left(\frac{1.26 \times 10^{-9}}{8.91 \times 10^{-10}}\right) \times 100 = 141\%$$

This result makes sense because pH 8.90 represents a higher hydrogen concentration than pH 9.05.

11.3 The precision as %RPD is calculated in the routine way as the relative percent difference between two check standards. When sample duplicates are analyzed, the pH must first be converted to hydrogen ion concentration before the %RPD can be calculated:

$$\%RPD = \left|\frac{H_1 - H_2}{(H_1 + H_2)/2}\right| \times 100$$

where

H_1 = hydrogen ion concentration of replicate 1
H_2 = hydrogen ion concentration of replicate 2

EXAMPLE

Two aliquots of the same sample are analyzed and the pH measurements are 8.90 and 9.00. The %RPD is calculated:

$$H_{8.90} = antilog[-8\ .90] = 1.26 \times 10^{-9}$$
$$H_{9.00} = antilog[9.00] = 1.0 \times 10^{-9}$$
$$\%RPD = \left|\frac{(1.26 \times 10^{-9}) - (1.00 \times 10^{-9})}{(1.26 \times 10^{-9} + 1.0 \times 10^{-9})/2}\right| \times 100 = \left|\frac{0.26 \times 10^{-9}}{1.13 \times 10^{-9}}\right| \times 100 = 23\%$$

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The pH meter/electrode will be calibrated using two pH buffers that bracket the expected range of pH.

12.2 Duplicate sample measurements will be performed on 10% of the samples.

12.3 A check solution from a separate source will be analyzed after the calibration as an initial calibration verification. A control limit of the theoretical pH ± 0.2 pH units will be used. This is equivalent to the pH recovery range of 63-158% over the pH range of 2 - 13. After every ten samples a check solution buffer

will be analyzed as a continuing calibration verification. The pH reading should be ±0.2 pH units of the true value. An RPD control limit of 40% will be used to gauge the acceptability of sample replicates.

13.0 PREVENTIVE MAINTENANCE

No additions to this revision.

14.0 TROUBLESHOOTING

No additions to this revision.

15.0 REFERENCES

1. *Savannah Laboratories' Comprehensive Quality Assurance Plan* and *Savannah Laboratories' Corporate Quality Assurance Plan*, current revisions.

2. EPA Method 150.1; *Methods for Chemical Analysis of Water and Wastes*, EPA 600/4-79-020.

3. SW-846 Methods 9040 and 9045C; *Test Methods for Evaluating Solid Waste, SW-846 Third Edition*.

4. Standard Method 4500-H^+; *Standard Method for the Examination of Water and Wastewater*, 18th Edition.

BA70 - pH

NEW ORLEANS

1. *In [the] procedure sequence, why is the pH 7.0 buffer checked and not the check solution (ERA, etc)?*

> The sequence will be changed - check solution should be where pH 7.0 buffer is.

2. *Where did you find that you had to convert the pH to [H^+] to calculate accuracy and precision?*

> See the definition in Section 1.2 of the DRAFT SOP. It should be clear that a 1 pH unit change is the result of a 10 fold change in [H^+]. For example, calculate the % RPD between pH 6 and pH 7 using "pH units."

$$\%RPD = = \frac{|7 - 6|}{(7 + 6)/2} = \frac{1}{6.5} = 15.4\%$$

Now calculate the %RPD between the [H^+]:

$$\%RPD = \frac{|1 \times 10^{-6} - 1 \times 10^{-7}|}{(1 \times 10^{-6} + 1 \times 10^{-7})/2} \times 100 = 164\%$$

3. *Can we mix organic liquids with water to get a pH to meet the corrosivity requirement? It is my understanding that you cannot measure the pH of non-aqueous liquids (organics).*

> Method 9045C indicates that mixing non-aqueous samples with water is the procedure for determining the pH. The sample prep section is taken directly from 9045C. The answer to the question is "NO" - 9045C cannot be used to determine whether a waste is corrosive. Method 1110 must be used for non-aqueous wastes.

4. *Should samples be allowed to equilibrate to room temperature before analysis?*

> Yes. It may be helpful to pour a well-mixed aliquot into a labeled container to make the sample warm up faster. The temperature of the sample should be ±2C of buffers.

5. *These ranges need to be incorporated into the CQAP.*

> Right. I will get a change notice out to you all as soon as I can.

6. *We do not have buffers lower than 4 or higher than 10. We will need to order these if this is incorporated.*

> Suggest pH 2.0 and 13.0.

MOBILE

7. *If we have limited soil, what do we do?*

> Text will be revised to allow smaller weights of sample and volumes of reagent water as long as the 1:1 ratio is maintained.

8. *What about checking pH for liquid samples for SVOC in extractions [lab]? It is not feasible to do electrode for samples set up for Method 3520 or herbicides.*

> pH paper is acceptable for measuring the pH ranges necessary for extractions.

TAMPA

9. *Section 10.3 Orion recommends not drying electrode.*

> Text will be modified.

10. *Section 10.1.2: Orion pH meter auto calibrates. Section 10.2 Orion pH meter does not have the ability to bracket the sample. It only standardizes on 7 and 4 or 7 and 10.*

> Most pH meters standardize on two pH buffers. The slope of the meter/electrode can be checked at a third pH to demonstrate that the meter gives a proportional response over the working range. (Section 10.1).

TALLAHASSEE

11. *Include the statement "pH paper can be used to determine the approximate pH. It can be used to check for proper preservative or if a sample is in the proper pH range preparation steps," in the SUMMARY OF METHOD section.*

> Good idea Will put this statement in SOP.

12. *Section 5.0 - The last sentence should read "The pH **must** be determined as soon as possible after collection." Delete last sentence in DRAFT.*

> OK. Text will be revised.

13. *Add this statement to Section 10.2: "Samples which are organic solvents should be measured using pH paper to avoid damage to the electrode."*

> Is there a method somewhere for directly determining pH of an organic material? If mixed with water as in Section 9 of SOP, the organic phase may be decanted and the pH measured on the aqueous phase. pH paper may be appropriate if the solvent and water are miscible

14. *Section 12.3 - An RPD control limit of 40% will be used to gauge the acceptability of sample replicates.*

> A difference of 0.2 pH units is about a 40% RPD on a concentration [H+] basis. Most PEs usually have an acceptance range of greater than 0.2 pH units. I understand that there may be some difficulty explaining this to clients.

SAVANNAH

15. *Section 4.2 - "The temperature of the sample should be at the same temperature as the buffer solutions." How do we ensure this? Are we actually going to check the temperature?*

> Our pH meters are temperature compensated. The temperature of the sample should be near (±2C) the temperature of the buffers (room temperature). No, we are not going to actually check the temperature. (See question 4).

16. *Sequence in Section 10: Is this going to fly with auditors who want our calibration to bracket samples?*

> See note in Section 10.1.4

DEERFIELD

No comments

CHANGE -IN-PROGRESS ATTACHMENT

SOP Document No: GE40: 02.02.98:3

SOP Description: Total and Amenable Cyanide: Autoanalyzer Procedure

Approval Signature: R. Wayne Robbins

Title: Corporate QA Manager Date: 8/25/98

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

The following section has been revised to correct the calculation of the cyanide stock concentration.

Titrate each cyanide solution with 0.141N or 0.0141N silver nitrate. Titrate the solution drop-wise near the endpoint until one drop of the titrant changes the color from yellow to salmon-pink. Record all standardization information into the cyanide stock standard standardization log. Calculate the concentration of the cyanide stock standard:

$$CN, mg/L = \frac{Na \otimes Va}{Vx} \otimes 52.04 \frac{mg}{meq} \otimes \frac{1000mL}{1L}$$

where
Na = normality of the silver nitrate solution
Va = volume of the silver nitrate solution used in titration, mL
Vx = volume of cyanide stock standard titrated, mL

The concentration of the cyanide solution is the average of the three individual calculations:

$$CNavg = \frac{CN\ 1 + CN\ 2 + CN\ 3}{3}$$

Approval Signature: R. Wayne Robbins

R. Wayne Robbins
Title: Corporate QA Manager Date: 03/06/98

TOTAL AND AMENABLE CYANIDE: AUTOANALYZER PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of inorganic cyanide in water samples, wastewater, wastes, soils, or soil leachates. The samples are distilled prior to analysis using the procedures in SL SOP GE43: *MIDI Distillation of Water and Soils for the Determination of Cyanide*. The soil leaching procedure and the preparation of samples to determine the cyanide amenable to chlorination are also included in the SL SOP GE43.

1.2 The reporting limit (RL), the method detection limit(MDL), and the accuracy and precision limits are given in the current revisions of the SL *Comprehensive Quality Assurance Plan* and the *Corporate Quality Assurance Plan*.

2.0 SUMMARY OF METHOD

2.1 Cyanide, as hydrocyanic acid (HCN), is released by refluxing the sample with strong acid and distillation of the HCN into an absorber-scrubber containing NaOH solution. The cyanide ion in the absorbing solution is then determined colorimetrically at a wavelength of 570 nm.

In the colorimetric measurement, the cyanide is converted to cyanogen chloride (CNCl) by reaction with chloramine-T at pH <8 without hydrolyzing to the cyanate ion. After the reaction is complete, a colored complex is formed upon the addition of pyridine-barbituric acid reagent. The concentration of NaOH must be the same in the standards, the scrubber solutions, and any dilutions of the original scrubber solution, to obtain colors of comparable intensity.

2.2 This method is based on the guidance in SW-846 Method 9010B (Distillation) and 9012A and on EPA Methods 335.2 and 335.4. A method summary is included at the end of this SOP.

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 Cyanide is extremely poisonous. Although very high levels of cyanide rarely occur in environmental samples, the analyst must be careful when handling these potentially hazardous and toxic samples. The standards and reagents used in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.3 The analyst should be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in this procedure.The MSDS denote the type of hazard that each reagent poses and the safe handling instructions for these compounds.

4.0 INTERFERENCES

4.1 Carbonates dissolved in the distillate will be realeased as carbon dioxide in the colorimetric reaction. The carbon dioxide gas will cause air spikes and peak shifts. If carbonates are present in the distillate, add a small portion of hydrated lime (calcium hydroxide) to the distillate and shake the distillate to mix. Filter an aliquot of the treated sample for analysis.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Container	Aqueous: 250mL Plastic Soils/Solids: 250mL Plastic
Preservative	Aqueous: NaOH to pH>12 Soils/Solids: None
Storage	4C +/- 2C from collection to analysis
Hold Time	14 days from date sampled
Interference Checks	Aqueous: Residual chlorine, sulfide, nitrate/nitrite, preservation (pH)

The interference and preservation checks are described in SL SOP GE 43: *MIDI Distillation of Water and Soils for the Determination of Cyanide*. Samples that are analyzed by this procedure must be distilled prior to analysis.

6.0 APPARATUS AND MATERIALS

6.1 Traacs 800 autoanalyzer system or Lachat Quickchem 8000

6.2 Volumetric glassware-Class A, various sizes

6.3 Pipettes, volumetric and disposable

6.4 Autosampler cups or tubes

7.0 REAGENTS

The preparation of reagents must be documented in accordance with SL SOP AN44. Do not store reagents or standards in volumetric glassware.

7.1 Reagent water-lab-generated deionized water, ASTM type I or Type II

7.2 Sodium hydroxide(NaOH)-reagent grade. Record the date opened on the container..

7.3 Sodium hydroxide solution, 0.25 N: (Diluent solution) Dissolve 10 g NaOH pellets in 800 mL DI water. Cool to room temperature and dilute to 1000 mL with DI water. Transfer to a labeled plastic container for storage.

7.4 Barbituric acid (C4H4N2O5)-reagent grade.

7.5 Pyridine (C5H5N)-reagent grade.

7.6 Hydrochloric acid (HCl)-concentrated, reagent grade.

7.7 Pyridine-barbituric acid reagent : This solution must be prepared exactly as written and must be prepared under a hood.

Place a stir bar and 15.0g of barbituric acid into a 1000-mL volumetric flask. Wash the walls of the flask with approximately 100 mL of reagent water, swirling the flask to wet all of the barbituric acid. Add via a 100-mL glass graduated cylinder, 75.0 mL of pyridine. Place the volumetric flask on the stir plate and begin mixing. While mixing, add via a 25-mL graduated cylinder, 15.0mL of concentrated hydrochloric acid. Remove the magnetic stirring bar, bring to volume with reagent water. The solution should become clear when diluted to volume. Transfer this solution to an amber glass container for storage at 4C. For reference only: the pH of this solution should be 5.6 ± 0.2.

7.8 Potassium dihydrogen phosphate (KH2PO4)-reagent grade

7.9 Sodium hydrogen phosphate (Na2HPO4)-reagent grade.

7.10 Phosphate buffer: To about 800 mL of DI water contained in a 1000-mL volumetric flask, add 27.2 g KH_2PO_4 and 0.56 g Na_2HPO_4. Add a magnetic stirring bar and mix vigorously until the salts have dissolved. Remove the magnetic stirring bar. Dilute to 1000 mL with DI water. Store the solution in a plastic container. For reference only: the pH of this solution should be 5.2 ± .2.

7.11 Chloramine-T (1-CH3C6H4-4-SO2NClNa*3H2O)-reagent grade.

7.12 Chloramine-T reagent: Dissolve by swirling 2.0 g Chloramine-T in about 400 mL of DI water contained in a 500 mL volumetric flask. Bring to volume with DI water and mix until homogeneous. For reference only: the pH of this solution should be 8.9 ± 0.2. This reagent must be prepared daily.

7.13 Silver nitrate solution (AgNO3)-purchased at 0.141N.

7.14 Rhodanine indicator-reagent grade.

8.0 STANDARDS

The preparation of the cyanide standard must be tracked in accordance with SL SOP AN41: *Standard Material Traceability*. Do not store standards in volumetric glassware.

8.1 Cyanide Stock Standards

Two stock standards are prepared from different sources of cyanide. The potassium cyanide stock standard is used to prepare the calibration standards and the spiking solution for the LCS and MS. The sodium cyanide stock standard is used to prepare the initial and continuing verification standards.

8.1.1 Potassium cyanide(KCN)-reagent grade.

8.1.2 KCN stock standard, 1000 mgCN/L: Transfer 2.503 g of KCN and 2 g of NaOH into a 1-L volumetric flask. Add 400-500mL of reagent water and swirl the flask to dissolve the KCN and NaOH. Dilute to volume with reagent water and transfer the stock standard to a labeled plastic container.

8.1.3 Sodium cyanide(NaCN)-reagent grade

8.1.4 NaCN stock standard, 1000 mgCN/L : Transfer 1.885g of NaCN and 2 g of NaOH into a 1-L volumetric flask. Add 400-500mL of reagent water and swirl the flask to dissolve the NaCN and NaOH. Dilute to volume with reagent water and transfer the stock standard to a labeled plastic container.

8.1.5 Standardization of the Cyanide Stock Standards

The 1000mg/L cyanide stock standards (KCN and NaCN) are standardized each time an intermediate (10mg/l) is prepared.

8.1.5.1 Add three 25.0mL aliquots of the 1000mg/L cyanide stock standard into each of three 250mL Erlenmeyer flasks. Add 75mL of the 0.25 N dilution solution to each flask. Add 100mL of the 0.25 N dilution solution to a fourth Erlenmeyer flask to serve as a blank.

NOTE: It is important that the pH be above 12 or the indicator will not give a clear endpoint.

8.1.5.2 Add 2 drops of rhodanine indicator and a Teflon stir bar to each beaker and place the flask on a magnetic stirrer.

8.1.5.3 Titrate each cyanide solution with 0.141N silver nitrate. Titrate the solution drop-wise near the endpoint until one drop of the titrant changes the color from yellow to salmon-pink. Record all standardization information into the cyanide stock standard standardization log. Calculate the concentration of the cyanide stock standard:

$$CN\ , mg\ /\ L = \frac{Na \otimes Va}{Vx} \otimes 26.02\ mg\ /\ meq \otimes \frac{1000\ mL}{L}$$

where
Na = normality of the silver nitrate solution
Va = volume of the silver nitrate solution used in titration, mL
Vx = volume of cyanide stock standard titrated, mL

The concentration of the cyanide solution is the average of the three individual calculations:

$$CNavg = \frac{CN\ 1 + CN\ 2 + CN\ 3}{3}$$

8.2 Intermediate Standard Preparation

The intermediate standards are prepared from the cyanide stock standards (after standardization) according to the following equation:

$$Vstock = \frac{C\text{int} \otimes V\text{int}}{Cstock}$$

Where
Vstock = volume of stock standard required to prepare the intermediate standard, mL
Cstock = concentration of the cyanide stock standard, mg/L (use CNavg)
Cint = concentration of the intermediate standard to prepare, mg/L
Vint = volume of intermediate standard to prepare, mL

8.2.1 KCN cyanide intermediate standard, 10 mg/L: To a 100-mL volumetric flask, add Vstock mL stock cyanide solution and bring to volume with diluent solution.

8.2.2 NaCN cyanide intermediate standard, 10mg/L: To a 100-mL volumetric flask, add Vstock mL stock cyanide solution and bring to volume with diluent solution.

8.3 Cyanide Calibration and Verification Standards

8.3.1 Use the 10-mg/L KCN cyanide working standard to prepare calibration standards as shown below. Bring all calibration standards to final volume with diluent solution.

Standard Concentration (mg/L)	Volume of 10-mg/L CN Standard (mL)	Final Volume (mL)
0.5	5.0	100
0.3	3.0	100
0.1	1.0	100
0.07	0.70	100
0.04	0.40	100
0.01	0.10	100
Cal Blank	0.00	100

8.3.2 ICV/CCV and ICB/CCB

8.3.2.1 The ICV and the CCV are prepared from the NaCN intermediate cyanide standard at a concentration of 200ug/L. Add 2.0mL of the 10mg/L NaCN intermediate standard to a 100mL volumetric flask and dilute to volume with 0.25N NaOH (diluent solution).

8.3.2.2 The 0.25N NaOH diluent solution is used as the ICB and CCB.

9.0 SAMPLE PREPARATION

The sample preparation steps are given in SL SOP GE43:*MIDI Distillation of Water and Soils for the Determination of Cyanide*

10.0 PROCDURE

Store distillates in the dark in a refrigerator until ready to analyze the samples on the autoanalyzer. The distillate must be analyzed within 14 days of the date of collection. The analytical sequence is given in the SOP Summary.

10.1 Traacs 800 Procedure

10.1.1 Set up the autoanalyzer as shown in the Traacs manual. Fill water containers with fresh DI water. Put reagent lines into a 1-L beaker filled with DI water and 2 mL Brij-35. Clamp down pump platens. Start the autoanalyzer (refer to SOP GE110).

10.1.2 When a regular bubble pattern is achieved, place the WHT/WHT line into the buffer solution, the RED/RED line into the pyridine/barbituric acid solution, and the ORN/GRN line into the chloramine-T solution, and the GRN/GRN autosampler line into the 0.25N NaOH solution. Allow a lag time of at least 10 min to regain a stable bubble pattern and assure good reagent mix.

10.1.3 Determine the base and gain. A 0.5 mg/L CN standard should give a relative absorbance of approximately 0.43. Start the tray. (Operations and calculations are explained in SOP GE110.)

10.1.4 After a tray is completed, pump DI water containing 0.2% Brij-35 through all pump tubes to cleanse the system.

10.2 Lachat Procedure

10.2.1 Set up Manifold as shown in the Lachat manual. Fill water containers with fresh DI water.

10.2.2 Place the GRY/GRY line into the pyridine-barbituric acid reagent, the ORN/ORN line into the chloramine-T reagent, the other ORN/ORN line in the phosphate buffer reagent and the YLW/YLW line onto the carrier reagent.

10.2.3 Clamp down the pump platens and turn on the pump. Allow a lag time of 10 minutes to assure a good reagent mix.

10.2.4 Input data system parameters.

10.2.5 Place standards and samples into the autosampler and start analysis.

10.2.6 After a tray is complete, pump DI water through the manifold for 10 minutes.

11.0 CALCULATIONS

11.1 The cyanide concentration of liquid samples is calculated by the autoanalyzer. The concentration may be expressed as mg/L or ug/L. The calculation on the autoanalyzer assumes that the initial volume of sample and the final volume of the distillate are the same. If the sample is diluted at any step of the preparation or analysis, the dilution factor must be taken into account in the calculation.

11.2 The cyanide concentration of soil samples (direct distillation) is calculated by the following equation:

$$CN, mg/kg, dw = \frac{C \otimes F \otimes DF}{W \otimes solids}$$

where
C = concentration in the sample distillate, mg/L
F = final volume of the distillate, L
W = weight of sample distilled, kg
Solids = (percent solids)/100
DF = dilution factor (if distillate requires dilution)

11.3 The concentration of amenable cyanide for liquids is the total cyanide concentration minus the cyanide concentration of the sample after treatment with chlorine as detailed in SL SOP GE43.

11.4 The concentration of total cyanide and cyanide after treatment with chlorine in soils (see SL SOP GE43) is calculated as below

$$CN, mg/kg, dw = \frac{C \times F \times DF}{W \times solids}$$

Where
C = concentration in the sample distillate, mg/L
F = final volume of the extraction, L
W = weight of sample extracted, kg
Solids = (percent solids)/100
DF = dilution factor (if distillate required dilution)

The concentration of amenable cyanide is simply the total cyanide concentration minus the cyanide concentration of the sample after treatment with chlorine as described in SL SOP GE43.

11.5 Accuracy is expressed as percent recovery (%REC). The percent recovery for an LCS is calculated:

$$\%REC = \frac{Clcs}{Tlcs} \otimes 100$$

where
Clcs = concentration of the LCS determined from the preparation and analysis, mg/L or mg/kg
Tlcs = theoretical (true) concentration of the LCS, mg/L or mg/kg

$$\%REC = \frac{Cms - Csample}{Tms} \otimes 100$$

where
Cms = concentration of the MS determined from the preparation and analysis, mg/L or mg/kg
Csample = concentration of the unspiked sample, mg/L or mg/kg
Tms = theoretical (true) concentration of the LCS, mg/L or mg/kg

11.6 Precision is reported as the relative percent difference (%RPD) between two replicate samples or spiked sample. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(\%REC1 - \%REC2)}{\left(\frac{\%REC1 + \%REC2}{2} \right)} \right| \otimes 100$$

where
%REC1 = percent recovery of the MS
%REC2 = percent recovery of the or MSD

Note that the absolute value of the calculation is reported; that is, only positive values are reported.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

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.

13.0 PREVENTIVE MAINTENANCE

See Section 10 of the current SL QAP and the Traacs and Lachat manuals.

14.0 TROUBLE-SHOOTING

Each day prior to initial analysis and after shutdown, flush flow cell with DI water.

Before analysis, check all pump tubes to make sure none are collapsed.

If unable to establish a stable reagent baseline, try a water baseline. If unable to achieve a water baseline, change the pump tubes.

Traacs 800 Only - If unable to achieve a steady bubble pattern, check all joints for blockage, then clean heating bath and flow cell with 0.25 N NaOH using a syringe adapted to the joints. Rinse well with DI water.

15.0 REFERENCES

1. *Traacs 800 Method 802-86T:* Bran+Lubbe, Technicon Industrial Systems: Tarrytown, NY, February, 1986. Revised June, 1987.

2. Lachat *QuickChem Method 10-2004-00-1-A Determination of Cyanide in Water*; Revision Date: 6 June 1996.

3. *Test Methods for Evaluationg Solid Waste, Third Edition, SW-846* (including Update III); U.S. EPA Office of Solid Waste and Emergency Response; Washington, D.C.

4. *Methods for Chemical Analysis of Water and Wastes*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983.

CYANIDE METHOD SUMMARY

HOLD/STORAGE

Routine Container	Aqueous: 250mL Plastic Soils/Solids: 250-mL Plastic
Preservative	Aqueous: NaOH to pH>12 Soils/Solids: None
Storage	4C (less than 6C with no frozen samples) from collection to analysis
Hold Time	14 days from collection
Interference Checks	Aqueous: Residual chlorine, sulfide, nitrate/nitrite, preservation (pH)

SAMPLE PREPARATION

Aqueous: MIDI, Macro, or custom distillation apparatus with molar volumes consistent with sample size.
Soils/Solids: Direct distillation
Soils/Wastes: Basic leaching procedure followed by distillation in MIDI or Macro distillation apparatus

Matrix	Distillation	Maximum Sample Volume/Weight	Final volume of Distillate
Aqueous or Leachate	MIDI, Macro, or Custom	MIDI - 50mL Macro – 500mL	50ml 250ml
Soils/Solids	Macro, MIDI, or Custom	MIDI-1g Macro - 10g	50mL 250ml

ANALYTICAL SEQUENCE- (9010B/9012A,335.2,335.4)

Initial Calibration- 6 point
Initial Calibration Verification (ICV)
Initial Calibration Blank (ICB)
LCS, method blank, and 7 Sample Measurements
Continuing Calibration Verification (CCV)
Continuing Calibration Blank (CCB)
10 Sample Measurements
CCV
CCB
10 Sample Measurements
CCV
CCB

The sequence continues until all of the samples are analyzed or the calibration verification or calibration blank fail the acceptance criteria. The sequence must always end with CCV/CCB.

DISTILLATION BATCH- (9010B/9012A,335.2,335.4)

QC ITEM	Frequency
Method blank	Per batch
Distilled cal standards (also serve as the LCS)	2 per batch-70ug/L and 200ug/L
MS	5% of samples per matrix
MSD	5% of samples per matrix
Samples	Up to twenty samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL (6 point curve is routinely analyzed)	Before sample analysis and when continuing calibration verification fail. (see sequence)	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/- 10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)	After initial calibration and after every 10 sample measurements. (see sequence)	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL SL CQAP Table 5	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank	Per batch	< RL SL CQAP Table 5	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
Distilled standards-(LCS/LCSD)	Per batch	+/-10% of expected value	-Check calculations -Reanalyze distillate -AN02 Decision Matrix
MS/MSD	At a frequency of 5% of samples of the same matrix (one MS and one MSD per twenty samples)	SL CQAP Table 5	-Check calculations -Reanalyze distillate -AN02 Decision Matrix
Initial Demonstration of Capability	Per analyst	-linearity criteria -recovery of QCS within 90-110% -determination of MDL	-Reanalyze samples
MDL Study	-SL SOP CA90	-SL SOP CA90	-repeat MDL study

Approval Signature: R. Wayne Robbins

Title: Corporate QA Manager

Date: 5/1/98

TOTAL CYANIDE: AUTODISTILLATION PROCEDURE

1.0 SCOPE AND APPLICATION

This method is used to determine the concentration of cyanide in water, aqueous wastes and leachates. It is used to determine the concentration of both total cyanide and cyanide amenable to chlorination. Cyanide determined by this method has a practical quantitation limit of 0.010 mg/L in liquids and 1.0 mg/kg in soils.

2.0 SUMMARY OF METHOD

In this method, samples are automatically taken up by the autoanalyzer. Metal-cyanide complexes in the samples are dissociated by UV irradiation. The samples are then automatically distilled, buffered, pyridine/barbituric acid is added, and the absorbance of the resulting complex measured at 570 nm.

This method is based on SW-846 Method 9012(1) and EPA Method 335.3 (2), and the soil extraction procedure is based on SW-846 Method 9010A(1).

3.0 INTERFERENCES

Oxidizing interferences may be present in the samples, as indicated by a positive test on KI starch paper. If this type of interference is found, the field crew must reduce these substances by addition of sufficient ascorbic acid to result in a negative KI-starch paper test, plus an additional 0.6 g per liter of sample.

Sulfide is a positive interference in this procedure. Samples are checked for the presence of sulfide upon arrival in the laboratory with lead acetate paper, which indicates sulfide concentrations down to a level of 5-10 mg/L. If sulfide is found to be present by this test, cadmium carbonate, $CdCO_3$, is added to the sample until no more black CdS precipitate forms. Avoid adding an excess $CdCO_3$, as carbonate can hinder the analysis by forming bubbles on the cuvette walls or by causing timing problems with autoanalysis procedures. Filter the samples promptly to limit adsorption or coprecipitation.

Nitrate can be an interference in sufficiently high concentrations (> 10 mg/L) in the presence of certain types of organic matter. Addition of 50 mL of 0.4 N sulfamic acid to the distillation flask will eliminate the effects of this interference.

Thiocyanates may be considered an interference in this procedure, although often they are considered part of the total cyanide concentration.

4.0 APPARATUS AND MATERIALS

Traacs 800 autoanalyzer system, complete with UV lamp, cyanide manifold, and autodistillation system.

5.0 REAGENTS AND STANDARDS

Mixed acid: Carefully add 100 mL of 85% phosphoric acid and 20 mL of hypophosphorous acid to about 800 mL of DI water in a 1-L volumetric flask. Cool, dilute to volume with DI water, and mix thoroughly.

Sodium hydroxide, 0.05 N: Add 2.00 g of solid sodium hydroxide to 800 mL DI water in a 1-L volumetric flask. Dissolve and dilute to volume with DI water.

Potassium iodide starch test paper

Sodium hypochlorite: 5.25% (Clorox bleach)

Ascorbic acid (crystals)

Phosphate buffer, pH 5.2: Dissolve 27.2 g of potassium dihydrogen phosphate (KH_2PO_4) and 0.56 g disodium hydrogen phosphate (Na_2HPO_4) in about 800 mL of DI water. Dilute to 1 L with DI water and mix thoroughly. The pH of this solution should be 5.2 ± 0.1.

Chloramine T: Dissolve 0.17 g of Chloramine T in 80 mL DI water in a 100-mL volumetric flask. Dilute to volume with DI water. Prepare fresh daily.

Pyridine/barbituric acid: Weigh out 15.00 g of barbituric acid into a 1-L volumetric flask. Wash the sides of the flask with about 100 mL of DI water. Add 75 mL of pyridine and mix. Add 15 mL of concentrated hydrochloric acid, mix, and dilute to volume with DI water. Use a magnetic mixer to dissolve this solution completely. This reagent must be made exactly as stated above. This reagent should be prepared fresh monthly.

Sodium hydroxide solution, 0.25 N: Dissolve 10 g NaOH solid in 800 mL DI water. Cool. Dilute to 1 L with DI water.

Cyanide stock standard, 1000-mg/L: Dissolve 2 g of solid NaOH in about 800 mL DI water in a 1-L volumetric flask. Add 2.503 g of potassium cyanide (KCN), dissolve, and dilute to volume. Standardize by titrating with standard silver nitrate solution. This stock standard should be prepared fresh monthly.

Cyanide intermediate standard, 10-mg/L: Pipet 1 mL of cyanide stock standard into a 100-mL volumetric flask. Dilute to volume with DI water. Prepare fresh daily.

Cyanide working standards: Standards are prepared at concentrations of 10, 40, 70, 100, 300 and 500 µg/L by pipeting appropriate quantities of cyanide intermediate standard and diluting to 10 mL with 0.25 N NaOH solution. Prepare fresh daily.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Liquid samples are collected in 120-mL plastic bottles preserved with sodium hydroxide to pH >12 (blue dot) and cooled to 4°C. Soil samples are collected in widemouth jars and cooled to 4°C. Holding time for cyanide samples is 14 days from date of sampling.

Samples are checked for the presence of sulfide with lead acetate paper, as well as proper pH with pH paper, upon their arrival in the lab.

7.0 PROCEDURE

7.1 Amenable Cyanide

Transfer 100 mL or 10 g (or less, depending on cyanide concentration) of sample into 500 mL beaker and place on magnetic stirrer in a fume hood.

Using potassium iodide starch paper, test for residual chlorine (indicated by dark blue color on paper).

If paper is clear, add a dropperful of Clorox to the stirring sample. If the paper turns blue, enough bleach has been added. If the paper does not turn blue, add bleach until it does so.

Set timer for 1 h and check every 15 min with potassium iodide starch paper. Make sure enough Clorox is present to keep paper blue.

After 1 h, add approximately 0.5 g ascorbic acid to stirring liquid. Test with potassium iodide-starch paper until sample tests clear, then add 0.5 g excess ascorbic acid.

Sample is now ready to be analyzed. An untreated (total) sample is also analyzed. If necessary to eliminate interferences, the samples can be manually distilled.

The total cyanide minus the cyanide determined in the chlorinated sample is the amenable cyanide.

7.2 Total Cyanide

Soil and sludge samples are extracted according to method 9010A. A 5-g portion of the soil is weighed out and placed in a 120-mL plastic bottle. A 1-mL aliquot of 50% w/w NaOH solution and 99 mL of DI water is added to the sample. Samples are placed in a rotator and allowed to extract for 16 hours. The extracts are filtered and analyzed as liquid samples.

The instrument is set up as shown in Figure 1. Samples, sample extracts, and QC standards are set up according to the desired protocol. (Refer to Basic Traacs 800 Operating Procedure.) The relative absorbance of a 0.50 mg/L CN standard should be approximately 0.43. Traacs Method 882-90T(3) is followed for the analysis of the samples.

7.3 Calculations

The cyanide concentration of liquid samples is calculated by the autoanalyzer, which has input for dilution factors.

The cyanide concentration of soil samples is calculated by the following equation:

$$CN, mg/kg\ dw = \frac{CN, mg/L \times 0.100\ L}{dry\ wt\ fraction \times g\ sample} \; X\ 1000 \frac{g}{kg}$$

8.0 QUALITY CONTROL/QUALITY ASSURANCE

A calibration curve with a minimum of five points is analyzed daily.

The correlation coefficient of the calibration curve must be ≥ 0.995.

An independent calibration verification standard is analyzed immediately upon calibration (ICV)

after every 10 samples (CCV), and at the end of every run.

A blank is analyzed immediately after each calibration verification standard.

A matrix spike and a matrix spike duplicate are analyzed for every batch or every 20 samples, whichever is more frequent.

An extraction blank (sand and extraction fluid) is analyzed for every batch of extracted soil samples, and is considered the method blank for solid samples.

A spiked blank (sand, extraction fluid and spike) is analyzed in duplicate for every batch of extracted soil samples, and is considered the LCS for solid samples.

8.1 Operating Notes

1. Each day prior to initial analysis, flush flow cell with syringe containing DI water.

2. The oil bath unit temperature must be greater than 100° C before starting to pump reagents.

3. Sparging nitrogen flow should be set at approximately 60 (185-195 cc/min).

4. The temperature of the distillation unit should be 150° C ± 2° C during operation.

5. The distillation coil should be removed from the still and cleaned weekly with 10 mL of 0.25 N NaOH, then washed thoroughly with 30-50 mL of DI water.

6. The silicone oil inside the distillation coil should be replaced monthly. Siphon out old oil, wipe, clean and dry chamber completely with paper toweling, and refill with 100 mL of fresh silicone oil. In the event silicone oil is unavailable, mineral oil may be substituted.

7. If leakage occurs around the UV lamp, remove lamp holder and replace all joints within with Acidflex tubing. Seal all joints with epoxy. Make sure epoxy is dry before replacing lamp.

8. Do not allow surfactants to flow through the UV digester or the distillation unit.

9. The pH of waste from the flow cell should be 5.7.

9.0 REFERENCES

1. "Test Methods for Evaluating Solid Waste," Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986.

2. "Methods for Chemical Analysis of Water and Wastes"; U.S. EPA Office of Research and Development: Cincinnati, Ohio, March 1983.

3. "Traacs 800 Method 882-90T": Bran + Luebbe, Technicon Industrial Systems: Tarrytown, NY, March 1990.

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager Date: 03/06/98

MIDI DISTILLATION OF WATER AND SOILS FOR THE DETERMINATION OF CYANIDE

1.0 SCOPE AND APPLICATION

This procedure is used to prepare water, soil, and waste samples for the analysis of cyanide. This SOP contains the procedures for the determination of total and amenable cyanide in water and soils. The default procedure for total cyanide in soils is to distill soils in the same manner as liquids. If cyanide amenable to chlorination is requested in soils, the soil is subjected to an extraction procedure prior to the chlorination step and the total cyanide is determined concurrently in the soil leachate.

2.0 SUMMARY OF METHOD

2.1 A measured amount of sample is refluxed with strong acid in a specially designed distillation apparatus-the MIDI distillation apparatus. Cyanide, as hydrocyanic acid (HCN), is released and trapped in an absorber-scrubber containing a sodium hydroxide solution. The cyanide collected in the absorbing solution is determined colorimetrically.

NOTE: The MIDI digestion/distillation unit is used in place of the "macro" units described in the EPA methods. The MIDI digestion/distillation utilizes the same proportion of reagents and sample as in the "macro" units and yields comparable results.

2.2 When cyanide amenable to chlorination is to be determined, the sample is treated with residual chlorine (Clorox) for one hour. After one hour, the excess chlorine is destroyed and the sample is analyzed for cyanide. The cyanide amenable to chlorination is the difference between the total cyanide and the cyanide measured in the sample after treatment with chlorine.

NOTE: If cyanide amenable to chlorination is requested for a soil sample, the sample is extracted with a sodium hydroxide solution. A portion of the leachate is distilled and reported as the "extractable" cyanide and a portion of the leachate is treated with chlorine and distilled. The leaching procedure is required because the direct chlorination of the sample may solubilize metal cyanides that will not be recovered by the direct distillation of the soil.

2.3 This method is based on SW-846 Methods 9010B, and 9013, and EPA Methods 335.2 and 335.4.

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 Cyanide is extremely poisonous. Although very high levels of cyanide rarely occur in environmental samples, the analyst must be careful when handling these potentially hazardous and toxic samples. Do not add acid to a cyanide sample unless performing one of the preparation steps outlined in this procedure. Acid will cause the evolution of deadly hydrogen cyanide gas.

3.3 The preparation of samples for cyanide amenable to chlorination must be done under a well ventilated hood.

3.4 The standards and reagents used in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment must be used when preparing and using the standards and reagents.

3.5 The analyst must be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in this procedure. The MSDS contain guidance on the type of hazard that each reagent poses and the safe handling instructions for these materials.

3.6 Care must be taken when handling the distillation apparatus. Before handling glassware that has been in use, check the temperature to make sure that it is not hot. The distillation flask contains an acidic solution. Acids can cause skin burns and destroy unprotected clothing.

4.0 INTERFERENCES

4.1 Chlorine and sulfide may be present in samples. Chlorine will cause a negative interference. Sulfide will cause a positive interference with the colorimetric determination of cyanide. The samples must be screened for chlorine and sulfide upon arrival in the lab.

4.2 Nitrates can cause a positive interference when the nitrate concentration exceeds 10 mg/L and certain organic materials are present in the sample. The addition of 5mL of 0.4 N sulfamic acid to the sample at the time of distillation will eliminate the formation of cyanide from nitrates and organic materials. If nitrates are suspected to be present , samples should be screened for the presence of nitrates upon arrival in the lab.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Soil samples are collected in wide-mouth glass jars equipped with Teflon-lined caps. Soil samples are stored at 4° C (less than 6° C with no frozen samples) until time of analysis. The recommended holding time is 14 days from sample collection.

5.2 Liquid samples are collected in 250mL plastic containers and are preserved by the addition of NaOH to a pH > 12. Liquid samples are stored at 4° C (less than 6° C with no frozen samples) until time of analysis. The holding time is 14 days from the date of collection.

5.2.1 Upon arrival in the laboratory, the pH must be determined to ensure that the sample has been properly preserved. Pour a small volume of the well mixed sample into a small plastic cup and test the pH of the aliquot with narrow range pH paper. If the paper is > 12, the sample is properly preserved. If the pH is < 12, adjust the pH of the sample to > 12 by adding small aliquots of 50% NaOH to the sample container. Shake the sample container to mix the 50% NaOH with the sample. Check the pH of the sample again. Continue adding small aliquots of 50% NaOH to the sample until the pH is > 12.

NOTE: Do not add more preservative than 1% of the volume of the sample. For example, if the sample volume is 500mL, do not add more than 5mL of preservative.

5.2.2 Upon arrival in the laboratory, the sample should be checked for the presence of sulfides. Pour a small aliquot of the sample into a plastic cup. Test the aliquot with lead acetate paper. If the lead acetate paper turns black, add cadmium carbonate to the sample and shake the container to mix. Check the sulfide content again with the lead acetate paper. Continue adding cadmium carbonate to the sample until the lead acetate paper no longer turns black when exposed to the sample.

5.2.3 Upon arrival in the laboratory, the sample must be checked for the presence of residual chlorine. Pour a small aliquot of the sample into a plastic cup. Test the aliquot with potassium iodide starch paper. If the paper turns blue or black, add sodium arsenite to the sample and shake the container to mix. Check the residual chlorine content again with the potassium iodide starch paper. Continue adding sodium arsenite to the sample until the indicator paper no longer turns blue or black when exposed to the sample.

5.2.4 If nitrates are suspected, prior to analysis (but not necessarily upon receipt), samples should be screened for nitrates. Pour a small aliquot of the sample into a plastic cup. Test the aliquot with a Nitrate/Nitrite test strip. If the strip indicates the presence of Nitrate/Nitrite, add 5ml of sulfamic acid solution (described in section 7.7) to 50ml of sample. Note the addition is performed at the beginning of the distillation process.

6.0 APPARATUS AND MATERIALS

6.1 MIDI cyanide distillation apparatus, Model MCV103 or equivalent, with appropriate glassware

6.2 Vacuum Pump

6.3 Tygon tubing

6.4 Volumetric flasks

6.5 Volumetric pipettes

7.0 REAGENTS

The preparation of reagents are tracked according to SL SOP AN44. Do not store reagents in volumetric glassware.

7.1 Reagent water, lab-generated deionized water – See SL SOP AN35 for maintenance procedures for DI water system.

7.2 Clorox bleach (5.25% sodium hypochlorite)

7.3 Potassium iodide (KI)/ starch paper-used to indicate the presence of chlorine

7.4 Sodium arsenite ($NaAsO_2$): reagent grade, used to eliminate interferences from chlorine and to destroy the excess chlorine in the cyanide amenable to chlorination procedure.

7.5 Lead acetate paper-used to indicate the presence of sulfide

7.6 Cadmium carbonate ($CdCO_3$): reagent grade, used to eliminate interferences from sulfides.

7.7 Sulfamic acid (H_2NSO_3H): reagent grade, used to eliminate interferences from nitrates.

7.8 Sulfamic acid solution (0.4 N): Dissolve 40 g of sulfamic acid in a small volume of reagent water in a 1L volumetric flask. Dilute to volume with reagent water. Transfer the reagent to an appropriate container; e.g., a 1L bottle.

7.9 Sodium hydroxide (NaOH): reagent grade

CAUTION: Heat will be evolved as the sodium hydroxide is dissolved in the water. Sodium hydroxide solutions are caustic and will cause skin burns and destroy unprotected clothing.

7.10 Dilution solution (0.25 N sodium hydroxide): Dissolve 10 g of sodium hydroxide in 800 mL of reagent water in a 1L volumetric flask. Dilute to volume with reagent water. Transfer the reagent to a 1L **PLASTIC** container.

7.11 Sodium hydroxide solution (1.25 N): Dissolve 50 g of sodium hydroxide in reagent water in a 1L volumetric flask. Cool and dilute to volume with reagent water. Transfer the reagent to a 1L **PLASTIC** container. Do not store reagents in volumetric glassware.

7.12 Sodium hydroxide (50%): Measure 100 mL of reagent water into a 400mL beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Weigh out 100 g of sodium hydroxide into a plastic container. Add a small quantity of the sodium hydroxide from the container to the reagent water in the beaker on the magnetic stir plate. As the sodium hydroxide dissolves, add more of the sodium hydroxide from the beaker until all 100g has been added. Cool and transfer the reagent to a **PLASTIC** container.

7.13 Sulfuric acid (H_2SO_4): reagent grade, concentrated.

7.14 Sulfuric acid solution (1:1): Measure 500 mL of reagent water into a 2L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500 mL of concentrated sulfuric acid to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a 1L bottle. Do not store reagents in volumetric glassware.

CAUTION: Use extreme caution when preparing this solution. Heat will be evolved as the acid and water combine. This solution will cause skin burns and destroy unprotected clothing.

7.16 Magnesium chloride hexa-hydrate ($MgCl_2 \cdot 6H_2O$): reagent grade

7.17 Magnesium chloride solution: While stirring, add 510 g $MgCl_2 \cdot 6H_2O$ in 500 mL of reagent water in a 1L volumetric flask. After the salt dissolves, dilute to volume with reagent water. Transfer the reagent to a 1L bottle. Do not store reagents in volumetric glassware.

8.0 STANDARDS

Sock standards prepared from neat materials. Certificates of analysis or a statement of purity must be received with all neat compounds. All preparation steps must be in accordance with SL SOP AN41:*Standard Material Traceability*. Do not store standard solutions in volumetric glassware.

Potassium cyanide is used to prepare the calibration standards and the spiking solutions. The preparation and standardization of the cyanide solution is given in SL SOP GE40: *Total and Amenable Cyanide: Autoanalyzer Procedure.*

8.1 Cyanide stock standard – approximately 1000 mg/L , prepared from potassium cyanide. See SL SOP GE40 for the preparation and standardization of this stock solution. The KCN cyanide stock is used to prepare the undistilled calibration standards, the distilled calibration standards (also used as LCS), and the matrix spiking solution.

8.2 Preparation of the KCN Cyanide Intermediate Standards

The KCN intermediate is used to prepare the calibration standards (distilled and undistilled), and the spiking solutions. Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Cs \otimes Vs = Ci \otimes Vi$$

$$Vs = \frac{Ci \otimes Vi}{Cs}$$

where
Vs = volume of stock standard needed to prepare the spiking solution(mL)
Cs = concentration of stock solution(mg/L)
Ci = concentration of intermediate solution to prepare(mg/L)
Vi = volume of spiking solution to prepare(mL)

Preparation of the KCN Intermediate (10mg/L)

Transfer Vs (see above) mL of the KCN cyanide stock standard (approximately 1000mg/L) to a 100mL volumetric flask and dilute to volume with 0.25N NaOH. Two standards are prepared from the KCN and distilled to verify the efficiency of the distillation process. The distilled standards are also evaluated and reported as LCS.

9.0 SAMPLE PREPARATION

9.1 Chlorination Step for Cyanide Amenable to Chlorination

The steps for the chlorination of the samples must be performed under a properly functioning fume hood.

9.1.1 Transfer 100mL of a liquid sample or 100mL of a soil leachate (Section 9.2) to a 500-mL beaker. A smaller volume of sample or leachate can be used if the cyanide concentration is known to be high.

9.1.2 Place the beaker onto a magnetic stir plate in a fume hood. Add a Teflon stir bar to the beaker and stir the sample.

9.1.3 Check the pH of sample with pH paper. The pH of the sample must be maintained at >11 for the duration of the chlorination procedure.

9.1.4 Test the chlorine level in the sample using potassium iodide-starch paper. If the paper turns blue, residual chlorine is present in the sample. If the test paper is clear, add 0.5 mL of Clorox to the stirring sample. Test the sample again with the potassium iodide-starch paper. If the test paper turns blue, enough Clorox has been added. If the paper remains clear, add Clorox until the paper turns blue. An excess of chlorine must be maintained throughout this procedure.

9.1.5 Check the sample every 15 minutes with potassium iodide-starch paper and wide range pH paper.

If the KI paper is blue, check the sample again in 15 minutes. If the paper is clear, add a 0.5mL of Clorox to the sample and check the sample again with the test paper. Continue adding small volumes of Clorox until the test paper remains blue.

If the pH >11, check the sample again in 15 minutes. If the pH <11, add a few drops of 50% NaOH and check the pH again. Continue adding small aliquots of 50% NaOH until the pH remains >11.

9.1.6 Continue stirring the sample for a total of 1 hour, checking the chlorine level and pH every 15 minutes and adjusting as needed.

9.1.7 After 1 hour, add approximately 0.5g of sodium arsenite to the stirring sample. Test the sample with potassium iodide-starch paper. If the test paper remains white, add a second 0.5g portion of sodium arsenite to the sample. If the test paper turns blue, add sodium arsenite in 0.5g increments until the test paper remains white. Add approximately 0.5 g excess of sodium arsenite to the sample.

9.1.8 The sample is now ready to be distilled and analyzed.

9.2 Soil Leaching Procedure

The leaching procedure is the default preparation step for the determination of amenable cyanide in soils. Use the direct digestion/distillation of soils (9.3) for total cyanide.

The pH of the sample must be maintained at pH > 10 throughout the leaching procedure. In most cases, the addition of 200mL of 0.25N NaOH will be sufficient to maintain the pH.

9.2.1 Transfer 10g (+/- 0.5g) of a homogenized soil sample to a labeled, 250mL plastic container. Record the weight of the sample to the nearest 0.1g in the cyanide soil extraction log.

9.2.2 Add 200mL of 0.25N sodium hydroxide to the container. Cap the container and mix thoroughly. Check the pH with narrow range pH paper. If the pH is not greater than 10, add small aliquots of 50% NaOH to bring the pH above 10.

9.2.3 Securely cap the container and place the container in a rotary spinning device for 16 hours.

9.2.4 Remove the containers from the rotator and allow the samples to settle. Allow the sample leachate to settle and decant into a separate labeled container. Check the pH of the leachate. If the pH is greater than 10, the sample is ready for distillation. If the pH is less than 10, the leaching procedure is repeated with a smaller aliquot of solid.

NOTE: If the leachate pH is less than 10, the pH of the sample should be determined. If the sample pH is highly acidic, the sample is not likely to contain cyanide amenable to the leaching procedure.

9.3 Sample Distillation

The distillation batch for cyanide may include both soils and liquids. The same method blank and LCS (distilled standards) can be used for both matrices. MS/MSD are required at a frequency of 5% for all matrices.

9.3.1 Remove the samples from the storage refrigerator and allow the samples to come to room temperature. Complete as much of the distillation log as possible for each batch of samples that are being prepared.

9.3.2 Assemble the distillation apparatus as shown in the manufacturer's manual. Place 50 mL of 0.25 N NaOH into each absorber tube.

9.3.3 Add the samples to the distillation tubes.

9.3.2.1 Mix the liquid sample by inverting the container several times and transfer 50mL of a liquid sample, the cyanide-amenable-to-chlorination sample, or soil leachate to the distillation tube. Record the volume of sample distilled on the cyanide distillation log.

Add a 50mL aliquot of 0.25N NaOH into a distillation unit for the method blank.

Transfer two 50mL aliquots of a sample from the batch into each of two distillation units for the MS and MSD.

Note: If cyanide-amenable-to-chlorination is requested on a soil matrix, the total cyanide is determined on the soil leachate.

9.3.2.2 Homogenize soil samples by stirring with a stainless steel spatula. Add 1g of the well mixed soil sample to the distillation tube. Add 50mL of reagent water to the distillation tube. Record the weight of the sample to the nearest 0.1g on the cyanide distillation log.

-Transfer two 1g aliquots of a sample from the batch into each of two distillation units for the MS and MSD.

-The method blank is performed using only the reagents used for the distillation; that is, add 50mL of 0.25N NaOH to the empty distillation unit and assume a sample weight of 1.0g.

9.3.3 Add 0.5mL of the KCN cyanide intermediate solution (10mg/L) to each matrix spike (MS) and matrix spike duplicate(MSD) sample.

The concentration of cyanide added to the liquid sample is

$$\frac{0.5\,mL \otimes 10\,mg/L}{50\,mL} = \frac{0.0005\,L \otimes 50\,mg/L}{0.050\,L} = 0.10\,mg/L = 100ug/L$$

The concentration of cyanide added to the soil/solid sample is

$$\frac{0.5\,mL \otimes 10\,mg/L}{(1\,g)(solids)} = \frac{0.00005\,L \otimes 10\,mg/L}{(0.0010\,kg)(solids)} = 5.0\,mg/kg\,(if\ \%\ solids = 100)$$

where (solids) is the decimal equivalent of the percent solids

9.3.3.4 Every day samples are distilled, two calibration standards are prepared, distilled, and analyzed. The volumes of the KCN cyanide intermediate added to prepare the standards are:

$$\frac{0.35\,mL \otimes 10\,mg/L}{50\,mL} = \frac{0.00035\,L \otimes 10\,mg/L}{0.050\,L} = 0.070\,mg/L = 70ug/L$$

$$\frac{1.0\,mL \otimes 10\,mg/L}{50\,mL} = \frac{0.0010\,L \otimes 10\,mg/L}{0.050\,L} = 0.200\,mg/L = 200ug/L$$

9.3.4 Connect the glassware and the tubing. Turn on the pump and the condenser water. Inspect each unit to ensure that there are no leaks in the glassware or in the tubing.

9.3.5 Set the timer to 120 minutes and the heating block temperature to 125C.

9.3.6 Add 2mL of the magnesium chloride solution followed by 5mL of 1:1 sulfuric acid to each flask through the distillation head. Rinse the inlet tube with a small aliquot of reagent water.

9.3.7 If the sample contains nitrate, add 5mL of sulfamic acid solution.

9.3.8 After the samples have been distilled, turn off the pump and allow tubes to cool for approximately 20 minutes.

9.3.9 Pour the scrubber solution into a labeled 100mL storage container.

9.3.10 Store the distillate in the dark in the refrigerator until ready to perform the colorimetric analysis. The distillate must be analyzed before the 14-day holding time is up.

NOTE: The hold time is measured from day of collection and **not** from the day of distillation.

10.0 PROCEDURE

The analytical procedure for the determination of cyanide is given in SL SOP GE40.

11.0 CALCULATIONS

The calculations for the determination of cyanide are given in SL SOP GE40.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

SL SOP AN02: *Analytical Batching*, Table 13.1 of the SL QAP, and the SOP Summary provide guidance on evaluating QC and sample data

13.0 PREVENTATIVE MAINTENANCE

13.1 Check the pump to insure that there is sufficient oil for proper operation. Change the oil at a frequency specified by the manufacturer.

13.2 Inspect the glassware and tubing each before and after use for signs of wear and breakage. Pay particular attention to the ground glass joints.

13.3 Change the tubing and connectors if sample or reagents get into lines.

13.4 Check the temperature of the heater block annually.

13.5 Rinse the glassware thoroughly with reagent water to remove all traces of soap. Soap residue that remains in the glassware may cause foaming in the distillation apparatus. The glassware should be rinsed with 10% nitric acid after rinsing with water to remove traces of cyanide and basic active sites and rinsed with deionized water to remove the acid residue.

14.0 TROUBLE-SHOOTING

See manufacturers manual for guidance on repairing the instrument.

15.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste, Third Edition*, SW-846; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

2. *Methods for Chemical Analysis of Water and Wastes*; U.S.EPA Office of Research and Development: Cincinnati, OH, March, 1983.

CYANIDE METHOD SUMMARY

HOLD/STORAGE

Routine Container	Aqueous: 250mL Plastic Soils/Solids: 250-mL Plastic
Preservative	Aqueous: NaOH to pH>12 Soils/Solids: None
Storage	4C (less than 6C with no frozen samples) from collection to analysis
Hold Time	14 days from collection
Interference Checks	Aqueous: Residual chlorine, sulfide, nitrate/nitrite, preservation (pH)

SAMPLE PREPARATION

Aqueous: MIDI, Macro, or custom distillation apparatus with molar volumes consistent with sample size.
Soils/Solids: Direct distillation
Soils/Wastes: Basic leaching procedure followed by distillation in MIDI or Macro distillation apparatus

Matrix	Distillation	Maximum Sample Volume/Weight	Final volume of Distillate
Aqueous or Leachate	MIDI, Macro, or Custom	MIDI - 50mL Macro – 500mL	50ml 250ml
Soils/Solids	Macro, MIDI, or Custom	MIDI-1g Macro - 10g	50mL 250ml

ANALYTICAL SEQUENCE- (9010B/9012A,335.2,335.4)

Initial Calibration- 6 point
Initial Calibration Verification (ICV)
Initial Calibration Blank (ICB)
LCS, method blank, and 7 Sample Measurements
Continuing Calibration Verification (CCV)
Continuing Calibration Blank (CCB)
10 Sample Measurements
CCV
CCB
10 Sample Measurements
CCV
CCB

The sequence continues until all of the samples are analyzed or the calibration verification or calibration blank fail the acceptance criteria. The sequence must always end with CCV/CCB.

DISTILLATION BATCH- (9010B/9012A,335.2,335.4)

QC ITEM	Frequency
Method blank	Per batch
Distilled cal standards (also serve as the LCS)	2 per batch-70ug/L and 200ug/L
MS	5% of samples per matrix
MSD	5% of samples per matrix
Samples	Up to twenty samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL (6 point curve is routinely analyzed)	Before sample analysis and when continuing calibration verification fail. (see sequence)	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/- 10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)	After initial calibration and after every 10 sample measurements. (see sequence)	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL SL CQAP Table 5	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank	Per batch	< RL SL CQAP Table 5	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
Distilled standards-(LCS/LCSD)	Per batch	+/-10% of expected value	-Check calculations -Reanalyze distillate -AN02 Decision Matrix
MS/MSD	At a frequency of 5% of samples of the same matrix (one MS and one MSD per twenty samples)	SL CQAP Table 5	-Check calculations -Reanalyze distillate -AN02 Decision Matrix
Initial Demonstration of Capability	Per analyst	-linearity criteria -recovery of QCS within 90-110% -determination of MDL	-Reanalyze samples
MDL Study	-SL SOP CA90	-SL SOP CA90	-repeat MDL study