IEA, Inc.
3000 Weston Parkway
Cary, NC 27513

Phone 919-677-0090
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IEA
An Aquarion Company

December 9, 1996

US EPA RECORDS CENTER REGION 5
464660

Deb Drain
Montgomery Watson
4525 South Wasatch Blvd., Suite #200
Salt Lake City, Utah 84124

SUBJECT: ACS QAPP - Region V Comments

Dear Ms. Drain:

The following responses are provided to the comments submitted from Region V for the ACS QAPP:

29. SW-846 Method 8260A (Water and Soil) MSS01201.NC

A. Retention times are not routinely included in the SOP for this method, since the retention times can shift based on instrument, column and calibration. However, the current retention times for water and soil (instrument specific) are attached for your records (Attachment 1). This information is provided with each report package specific to the instrument and column in use at the time of analysis.

B. Table 2.0 of this SOP is attached for your records (Attachment 2).

30. SW-846 Method 8260A Low Concentration (Water) MSS01101.NC

Section 2.1 and Table 1.0

Retention times are not routinely included in the SOP for this method, since the retention times can shift based on instrument, column and calibration. However, the current retention times for water and soil (instrument specific) are attached for your records (Attachment 3). This information is provided with each report package specific to the instrument and column in use at the time of analysis.

32. GC/MS Method 8270 MSS00204.NC

A. Section 2.1 and Attachment A

Retention times are not routinely included in the SOP for this method, since the retention times can shift based on instrument, column and calibration. However, the

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current retention times for water and soil (instrument specific) are attached for your records (Attachment 4). This information is provided with each report package specific to the instrument and column in use at the time of analysis. The quantitation ions used are also included on the quantitation reports issued with the deliverable data package.

B. Section 10.6.1

The eight surrogates listed are all included in the surrogate spike mix as received from the vendor. This same surrogate spike mix is also used in the analysis of the CLP semivolatile BNA method where the additional two surrogate compounds are required. The additional compounds are not evaluated for the SW-846 Method 8270 analysis.

33. SOP for GC Method 8081 Pesticides + PCBs in Water

Although the SOP for GC Method 8080 Pesticides + PCBs in Water, GCS00900.NC was provided, the procedure to be used has now been determined as Method 8081. The SOP for this method is attached (Attachment 5). As noted above, the retention times will vary with instrument, column and/or calibration. For this reason, the specific retention times are not typically included in method SOPs due to the need to update these times. A table has been incorporated identifying the IEA minimum retention time requirements.

34. SOP for GC Method 8081 Pesticides + PCBs in Soil

A./B. Although the SOP for GC Method 8080 Pesticides + PCBs in Soil, GCS01000.NC was provided, the procedure to be used has now been determined as Method 8081. The SOP for this method is attached. As noted above, the retention times will vary with instrument, column and/or calibration. For this reason, the specific retention times are not typically included in method SOPs due to the need to update these times. Attached for your records are an example listing of the retention times for this method as it would be seen in reported data (Attachment 6).

C. Soil units in the 8081 SOP will be utilized, where applicable..

D. The standard clean up procedure required for the pesticide/PCB method is sulfur cleanup. The SOP for this procedure is attached (Attachment 7). Other cleanup procedures are not expected to be used for this project.

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36. SOP for TJA 61E Trace by Method 6010A MES02500.NC

A. Digestion procedures for aqueous samples have been attached. These include the SOPs for SW-846 Methods 3005, 3010 and 3015 (Attachments 8, 9 and 10 respectively).

B. Section 11.0

The SOP is written to incorporate the specific instrumental/software operating parameters necessary to accurately perform the method. It is unclear what other general instrumental parameters would be appropriate for the laboratory specific SOP. The recommended wavelengths for the project analytes have been added to the SOP (Table Three) and a copy of the revised SOP (MES02501.NC) has been attached for your files (Attachment 11).

C. Table Two, pg 10/10
Soil units have been added to the SOP, expressed as mg/Kg. Please note that actual PQLs for soils will vary based on the dry weight of the samples.

D. Table Two, pg 10/10
The PQLs listed in this SOP are the standard IEA devliverable PQL, however, MDL data indicates that the reporting limits for this project can also be met, eliminating any disparity with Attachment C, Table C-8.

37. SOP for Hexavalent Chromium in Soil CVS03902.NC

Section 13.0

The SOP has been revised (CVS03903.NC) to include corrective action (Section 10.6.1) for post-digestion spike recovery that does not meet acceptance criteria. Please see the revised document (Attachment 13).

39. SOP for Acidity Acidity.NC

Section 12.2

The calculation for MS % recovery has been added to the document in question and the revised SOP attached for your files (Attachment 12)

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Please note that every effort has been made to provide the revised SOPs as discussed above upon completion. Should you have any questions, do not hesitate to contact Mike McFadden or myself at 800-444-9919.

Very truly yours,

IEA, Inc.

Linda F. Mitchell
Director, QA

Attachments

cc: Jack Dullaghan
Keith Scott
Don Stogner

...\letter\clients\mongwat.sop

Monroe, Connecticut 203-261-4458

Schaumburg, Illinois 847-705-0740

N. Billerica, Massachusetts 508-667-1400

Whippany, New Jersey 201-428-8181

Laboratory Attachments

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ATTACHMENT 1

Monroe,
Connecticut
203-261-4458

Schaumburg,
Illinois
847-705-0740

N. Billerica,
Massachusetts
508-667-1400

Whippany,
New Jersey
201-428-8181

Quantitation Report R.T. 8260 Water 5mL purge

Data File : C:\HPCHEM\1\DATA\961119J.B\1119J01.D
Acq On : 19 Nov 96 3:00 pm
Sample : VSTD050 IEA MSD10
Misc : WATER LOW 1X
Quant Time: Nov 19 15:33 1996

Vial: 1
Operator: MOORE
Inst : MSD10
Dilution: 1.00

Method : C:\HPCHEM\1\DATA\961119J.B\8260AW.M
Title : 8260A IEA MSD10
Last Update : Tue Nov 19 13:54:18 1996
Response via : Multiple Level Calibration

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) Pentafluorobenzene	9.69	168	2685761	50.00	µg/L	0.00
11) 1,4-Difluorobenzene	11.21	114	2502952	50.00	µg/L	0.00
37) Chlorobenzene-d5	17.56	117	2243450	50.00	µg/L	0.00
62) 1,4-Dichlorobenzene-d4	23.05	152	1370420	50.00	µg/L	0.00
System Monitoring Compounds						%Recovery
14) 4-Bromofluorobenzene	20.30	95	1378360	46.99	µg/L	93.97%
26) 1,2-Dichloroethane-d4	10.23	102	230348	48.20	µg/L	96.40%
35) Toluene-d8	14.35	98	2335179	48.56	µg/L	97.12%
Target Compounds						Qvalue
2) Acrylonitrile	6.34	53	99157	45.28	µg/L	94
3) Allyl chloride	5.48	76	236180	53.06	µg/L	93
4) Dichlorodifluoromethane	2.30	85	536714	45.71	µg/L	99
5) 2,2-Dichloropropane	8.45	77	1237150	47.22	µg/L	100
6) Iodomethane	5.00	142	1181943	43.51	µg/L	97
7) Methacrylonitrile	9.02	41	220296	47.14	µg/L	97
8) Methyl-tert-Butyl ether	6.38	73	1536390	46.43	µg/L	99
9) Trichlorofluoromethane	3.78	101	1251830	48.83	µg/L	100
10) Vinyl acetate	7.43	43	754455	44.79	µg/L	97
12) Acetone	4.92	43	91998	47.06	µg/L	91
13) Bromochloromethane	8.99	128	410132	53.17	µg/L	97
15) Bromomethane	3.19	94	314511	49.88	µg/L	99
16) 2-Butanone	8.62	43	163870	42.02	µg/L	92
17) Carbon disulfide	5.10	76	1285810	48.42	µg/L	100
18) Chloroethane	3.35	64	167642	56.23	µg/L	99
19) 2-Chloroethyl vinyl ether	13.53	63	330799	47.77	µg/L	99
20) Chloroform	9.20	83	1511605	49.89	µg/L	99
21) Chloromethane	2.56	50	226532	46.00	µg/L	99
22) Dibromomethane	12.44	93	794181	49.01	µg/L	98
23) 1,1-Dichloroethane	7.23	63	987706	47.87	µg/L	100
24) 1,2-Dichloroethane	10.39	62	940780	48.54	µg/L	98
25) 1,1-Dichloroethene	4.72	96	550105	49.12	µg/L	96
27) cis-1,2-Dichloroethene	8.50	96	723466	49.60	µg/L	98
28) trans-1,2-Dichloroethene	6.33	96	652919	49.10	µg/L	96
29) 1,2-Dichloroethene (total)	8.50	96	723466	49.60	µg/L	97
30) 1,3-Dichloropropane	15.77	76	1157276	51.12	µg/L	98
31) 1,1-Dichloropropene	9.89	75	986425	51.18	µg/L	98
32) Ethyl methacrylate	15.29	69	924730	47.28	µg/L	99
33) Methylene chloride	5.75	84	538439	48.45	µg/L	96
34) Methyl methacrylate	12.56	69	448077	46.47	µg/L	99
36) Vinyl chloride	2.71	62	282787	48.69	µg/L	100
38) Benzene	10.32	78	1625957	44.91	µg/L	100
39) Bromodichloromethane	12.82	83	1648256	43.33	µg/L	100

(#) = qualifier out of range (m) = manual integration

Quantitation Report

Data File : J:\HPCHEM\1\DATA\961119J.B\1119J01.D Vial: 1
Acq On : 19 Nov 96 3:00 pm Operator: MOORE
Sample : VSTD050 IEA MSD10 Inst : MSD10
Misc : WATER LOW 1X Dilution: 1.00
Quant Time: Nov 21 8:36 1996

Method : J:\HPCHEM\1\DATA\961119J.B\8260AW.M
Title : 8260A IEA MSD10
Last Update : Tue Nov 19 15:37:48 1996
Response via : Multiple Level Calibration

	Compound	R.T.	QIon	Response	Conc Unit		Qvalue
40)	Carbon tetrachloride	9.88	117	1536535	43.88 µg/L		99
41)	Chlorodibromomethane	16.26	129	1783772	44.56 µg/L		100
42)	Chlorobenzene	17.62	112	2066186	50.33 µg/L		99
43)	1,2-Dibromoethane	16.49	107	1393070	44.93 µg/L		100
44)	cis-1,4-Dichloro-2-butene	20.17	75	75389	15.26 µg/L		98
45)	trans-1,4-Dichloro-2-buten	20.86	53	114213	18.06 µg/L		90
46)	1,2-Dichloropropane	12.19	63	616287	44.36 µg/L		98
47)	cis-1,3-Dichloropropene	13.79	75	1135833	45.26 µg/L		100
48)	trans-1,3-Dichloropropene	15.02	75	1074993	43.96 µg/L		100
49)	Ethylbenzene	17.90	91	3036335	50.38 µg/L		99
50)	2-Hexanone	16.05	43	272444	34.96 µg/L		90
51)	4-Methyl-2-pentanone	14.17	43	514500	41.22 µg/L	#	91
52)	Styrene	19.13	104	2062298	49.31 µg/L		99
53)	1,1,1,2-Tetrachloroethane	17.82	131	1253104	49.18 µg/L		98
54)	Tetrachloroethene	15.69	164	1919302	50.30 µg/L		99
55)	Toluene	14.49	91	2340429	44.88 µg/L		99
56)	1,1,1-Trichloroethane	9.54	97	1543111	43.93 µg/L		98
57)	1,1,2-Trichloroethane	15.42	97	840729	44.70 µg/L		99
58)	Trichloroethene	11.73	95	1154966	47.37 µg/L		99
59)	m,p-Xylene	18.18	106	2402778	100.60 µg/L		98
60)	o-Xylene	19.08	106	1216517	49.67 µg/L		100
61)	Xylene (total)	19.08	106	1216517	49.69 µg/L		99
63)	Bromoform	19.52	173	1467967	49.42 µg/L		99
64)	Bromobenzene	20.62	156	1162893	48.99 µg/L		99
65)	n-Butylbenzene	24.02	91	2634532	49.95 µg/L		99
66)	tert-Butylbenzene	22.13	119	2928753	47.54 µg/L		98
67)	sec-Butylbenzene	22.67	105	3637909	48.13 µg/L		100
68)	n-Propylbenzene	20.94	91	3180572	47.52 µg/L		99
69)	2-Chlorotoluene	21.09	91	2226164	48.41 µg/L		99
70)	4-Chlorotoluene	21.37	91	2503645	48.35 µg/L		96
71)	1,2-Dibromo-3-chloropropan	25.74	75	306856	41.28 µg/L		99
72)	1,2-Dichlorobenzene	23.97	146	1978411	49.47 µg/L		98
73)	1,3-Dichlorobenzene	22.88	146	1985399	47.60 µg/L	m	97
74)	1,4-Dichlorobenzene	23.10	146	2169222	49.56 µg/L		97
75)	Hexachlorobutadiene	27.27	225	1061309	50.15 µg/L		100
76)	p-Isopropyltoluene	23.03	119	2896307	47.80 µg/L		99
77)	Isopropylbenzene	19.96	105	3176423	51.40 µg/L		100
78)	Naphthalene	27.35	128	2954955	47.13 µg/L		100
79)	Pentachloroethane	22.15	167	142505	26.72 µg/L		98
80)	1,1,2,2-Tetrachloroethane	20.70	83	828146	43.33 µg/L		100
81)	1,2,4-Trichlorobenzene	27.00	180	1778094	48.50 µg/L		99
82)	1,2,3-Trichlorobenzene	27.73	180	1687700	48.24 µg/L		98
83)	1,2,3-Trichloropropane	20.77	75	955665	45.24 µg/L		99
84)	1,3,5-Trimethylbenzene	21.38	105	2452876	48.71 µg/L		97

(#) = qualifier out of range (m) = manual integration

Quantitation Report

Data File : C:\HPCHEM\1\DATA\961119J.B\1119J01.D
Acq On : 19 Nov 96 3:00 pm
Sample : VSTD050 IEA MSD10
Misc : WATER LOW 1X
Quant Time: Nov 19 15:33 1996

Vial: 1
Operator: MOORE
Inst : MSD10
Dilution: 1.00

Method : C:\HPCHEM\1\DATA\961119J.B\8260AW.M
Title : 8260A IEA MSD10
Last Update : Tue Nov 19 13:54:18 1996
Response via : Multiple Level Calibration

Compound	R.T.	QIon	Response	Conc Unit	Qvalue
85) 1,2,4-Trimethylbenzene	22.26	105	2645944	47.84 µg/L	100

(#) = qualifier out of range (m) = manual integration

Quantitation Report RT. Low Soil 8260

Data File : C:\HPCHEM\1\DATA\961108G.B\1108G01.D Vial: 15
Acq On : 8 Nov 96 7:15 pm Operator: CREWES
Sample : VSTD050 IEA MSD7 Inst : MSD7
Misc : SOIL LOW 1X Dilution: 1.00
Quant Time: Nov 8 19:54 1996

Method : C:\HPCHEM\1\DATA\961108G.B\8260AS.M
Title : 8260A SOIL
Last Update : Fri Nov 08 20:07:26 1996
Response via : Multiple Level Calibration

Internal Standards	R.T.	QIon	Response	Conc Units	Dev(Min)
1) Pentafluorobenzene	10.18	168	1417186	50.00 ug/kg	0.00
11) 1,4-Difluorobenzene	11.70	114	1303627	50.00 ug/kg	0.02
37) Chlorobenzene-d5	18.10	117	1102966	50.00 ug/kg	-0.02
62) 1,4-Dichlorobenzene-d4	23.63	152	772191	50.00 ug/kg	0.00
System Monitoring Compounds					%Recovery
14) 4-Bromofluorobenzene	20.87	95	1139296	52.59 ug/kg	105.18%
26) 1,2-Dichloroethane-d4	10.73	102	123725	51.72 ug/kg	103.44%
35) Toluene-d8	14.89	98	1309504	46.83 ug/kg	93.65%
Target Compounds					Qvalue
2) Acrylonitrile	6.88	53	80853	45.23 ug/kg#	90
3) Allyl chloride	6.06	76	154753	45.10 ug/kg	99
4) Dichlorodifluoromethane	2.59	85	650476	58.65 ug/kg	98
5) 2,2-Dichloropropane	8.99	77	1224216	59.85 ug/kg#	79
6) Iodomethane	5.55	142	963050	52.75 ug/kg	99
7) Methacrylonitrile	9.53	41	195002	50.56 ug/kg	97
8) Methyl-tert-Butyl ether	6.95	73	1356433	53.30 ug/kg	98
9) Trichlorofluoromethane	4.25	101	1451672	63.93 ug/kg	100
10) Vinyl acetate	7.99	43	901306	51.07 ug/kg	99
12) Acetone	5.46	43	144773	60.78 ug/kg	99
13) Bromochloromethane	9.51	128	252462	51.25 ug/kg	98
15) Bromomethane	3.59	94	440016	49.65 ug/kg	99
16) 2-Butanone	9.13	43	171384	52.05 ug/kg#	81
17) Carbon disulfide	5.67	76	898292	45.04 ug/kg	99
18) Chloroethane	3.76	64	197440	43.57 ug/kg	97
19) 2-Chloroethyl vinyl ether	14.05	63	180314	35.21 ug/kg	98
20) Chloroform	9.71	83	1455668	58.71 ug/kg	100
21) Chloromethane	2.89	50	132576	45.93 ug/kg	94
22) Dibromomethane	12.96	93	586244	54.84 ug/kg	96
23) 1,1-Dichloroethane	7.79	63	917803	51.94 ug/kg	99
24) 1,2-Dichloroethane	10.88	62	1177729	64.88 ug/kg	99
25) 1,1-Dichloroethene	5.29	96	389752	49.63 ug/kg	92
27) cis-1,2-Dichloroethene	9.01	96	494467	49.00 ug/kg	97
28) trans-1,2-Dichloroethene	6.88	96	445297	49.93 ug/kg	93
29) 1,2-Dichloroethene (total)	9.01	96	494467	49.00 ug/kg	96
30) 1,3-Dichloropropane	16.33	76	804563	51.92 ug/kg	99
31) 1,1-Dichloropropene	10.38	75	892690	58.23 ug/kg	100
32) Ethyl methacrylate	15.84	69	641765	48.09 ug/kg	98
33) Methylene chloride	6.30	84	389330	48.35 ug/kg	100
34) Methyl methacrylate	13.07	69	303431	47.15 ug/kg	99
36) Vinyl chloride	3.06	62	218271	47.52 ug/kg	99
38) Benzene	10.81	78	1219270	52.38 ug/kg	100
39) Bromodichloromethane	13.33	83	1342107	57.46 ug/kg	97

(#) = qualifier out of range (m) = manual integration

Quantitation Report

Data File : C:\HPCHEM\1\DATA\961108G.B\1108G01.D Vial: 15
Acq On : 8 Nov 96 7:15 pm Operator: CREWES
Sample : VSTD050 IEA MSD7 Inst : MSD7
Misc : SOIL LOW 1X Dilution: 1.00
Quant Time: Nov 8 19:54 1996

Method : C:\HPCHEM\1\DATA\961108G.B\8260AS.M
Title : 8260A SOIL
Last Update : Fri Nov 08 20:07:26 1996
Response via : Multiple Level Calibration

	Compound	R.T.	QIon	Response	Conc	Unit	Qvalue
40)	Carbon tetrachloride	10.36	117	1416165	63.85	ug/kg	98
41)	Chlorodibromomethane	16.83	129	1103170	53.35	ug/kg	99
42)	Chlorobenzene	18.19	112	1142929	51.49	ug/kgm	1
43)	1,2-Dibromoethane	17.05	107	820318	52.94	ug/kg	99
44)	cis-1,4-Dichloro-2-butene	20.72	75	69880	18.56	ug/kg	96
45)	trans-1,4-Dichloro-2-buten	21.42	53	156529	26.35	ug/kg	88
46)	1,2-Dichloropropane	12.70	63	392561	48.18	ug/kg#	98
47)	cis-1,3-Dichloropropene	14.31	75	763405	52.12	ug/kg	96
48)	trans-1,3-Dichloropropene	15.57	75	812102	52.65	ug/kg	100
49)	Ethylbenzene	18.44	91	1996500	54.58	ug/kg	100
50)	2-Hexanone	16.59	43	279554	50.86	ug/kg	99
51)	4-Methyl-2-pentanone	14.73	43	398994	49.66	ug/kg	99
52)	Styrene	19.69	104	1109851	50.06	ug/kg	99
53)	1,1,1,2-Tetrachloroethane	18.40	131	732420	52.04	ug/kg	98
54)	Tetrachloroethene	16.24	164	589221	52.35	ug/kg	100
55)	Toluene	15.02	91	1339620	50.85	ug/kg	98
56)	1,1,1-Trichloroethane	10.03	97	1459574	63.60	ug/kg	99
57)	1,1,2-Trichloroethane	15.96	97	465786	49.46	ug/kg	98
58)	Trichloroethene	12.23	95	667306	53.55	ug/kg	95
59)	m,p-Xylene	18.74	106	1289381	99.25	ug/kg	98
60)	o-Xylene	19.65	106	644768	49.74	ug/kg	97
61)	Xylene (total)	19.65	106	644768	49.74	ug/kg	97
63)	Bromoform	20.09	173	772944	49.16	ug/kg	99
64)	Bromobenzene	21.19	156	663950	51.33	ug/kg#	24
65)	n-Butylbenzene	24.61	91	2122047	56.61	ug/kg	98
66)	tert-Butylbenzene	22.72	119	2379367	54.76	ug/kg	91
67)	sec-Butylbenzene	23.25	105	2426483	55.58	ug/kg	100
68)	n-Propylbenzene	21.52	91	2431100	55.57	ug/kg	96
69)	2-Chlorotoluene	21.67	91	1713699	56.12	ug/kg	93
70)	4-Chlorotoluene	21.93	91	2111522	57.07	ug/kg	97
71)	1,2-Dibromo-3-chloropropan	26.12	75	332630	53.53	ug/kg	98
72)	1,2-Dichlorobenzene	24.56	146	1184747	53.87	ug/kg	98
73)	1,3-Dichlorobenzene	23.46	146	1194470	54.32	ug/kg	98
74)	1,4-Dichlorobenzene	23.69	146	1308355	55.30	ug/kg	98
75)	Hexachlorobutadiene	27.65	225	777077	59.49	ug/kg	98
76)	p-Isopropyltoluene	23.61	119	2136403	57.31	ug/kg	100
77)	Isopropylbenzene	20.53	105	1979431	56.71	ug/kg	99
78)	Naphthalene	27.76	128	1619684	51.59	ug/kg	100
79)	Pentachloroethane	22.73	167	552307	51.53	ug/kg	98
80)	1,1,2,2-Tetrachloroethane	21.26	83	868849	53.57	ug/kg	98
81)	1,2,4-Trichlorobenzene	27.38	180	947764	56.93	ug/kg	98
82)	1,2,3-Trichlorobenzene	28.17	180	909795	56.76	ug/kg	99
83)	1,2,3-Trichloropropane	21.33	75	828365	53.63	ug/kg	92
84)	1,3,5-Trimethylbenzene	21.94	105	1844542	56.71	ug/kg	100

(#) = qualifier out of range (m) = manual integration

Quantitation Report

Data File : C:\HPCHEM\1\DATA\961108G.B\1108G01.D Vial: 15
Acq On : 8 Nov 96 7:15 pm Operator: CREWES
Sample : VSTD050 IEA MSD7 Inst : MSD7
Misc : SOIL LOW 1X Dilution: 1.00
Quant Time: Nov 8 19:54 1996

Method : C:\HPCHEM\1\DATA\961108G.B\8260AS.M
Title : 8260A SOIL
Last Update : Fri Nov 08 20:07:26 1996
Response via : Multiple Level Calibration

Compound	R.T.	QIon	Response	Conc Unit	Qvalue
85) 1,2,4-Trimethylbenzene	22.83	105	1944588	56.67 ug/kg	97

(#) = qualifier out of range (m) = manual integration

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ATTACHMENT 2

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TABLE 2

PRACTICAL QUANTITATION LIMITS FOR 8260A VOLATILE ANALYSIS

ANALYTE	PRACTICAL QUANTITATION LIMIT (PQL)(ug/L or ug/Kg)
Acetone	50
Acrylonitrile	5
Allyl Chloride	5
Benzene	5
Bromobenzene	5
Bromochloromethane	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	10
2-Butanone	10
n-Butylbenzene	5
tert-Butylbenzene	5
sec-Butylbenzene	5
Carbon Disulfide	5
Carbon Tetrachloride	5
Chlorobenzene	5
Chlorodibromomethane	5
Chloroethane	10
2-Chloroethylvinyl Ether	10
Chloroform	5
Chloromethane	10
2-Chlorotoluene	5
4-Chlorotoluene	5
1,2-Dibromo-3-Chloropropane	5
1,2-Dibromoethane	5
Dibromomethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
Dichlorodifluoromethane	10
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
cis-1,2-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
1,3-Dichloropropane	5
2,2-Dichloropropane	5
1,1-Dichloropropene	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
cis-1,4-Dichloro-2-Butene	5

TABLE 2 (Continued)

PRACTICAL QUANTITATION LIMITS FOR 8260A VOLATILE ANALYSIS

ANALYTE	PRACTICAL QUANTITATION LIMIT (PQL)(ug/L or ug/Kg)
trans-1,4-Dichloro-2-Butene	5
Ethyl Benzene	5
Ethyl Methacrylate	5
Hexachlorobutadiene	5
2-Hexanone	10
Iodomethane	5
Isopropylbenzene	5
p-Isopropyltoluene	5
Methacrylonitrile	5
Methylene Chloride	10
Methyl Methacrylate	5
4-Methyl-2-Pentanone	10
Methyl-tert-Butyl Ether	5
Naphthalene	5
Pentachloroethane	5
n-Propylbenzene	5
Styrene	5
1,2,3-Trichlorobenzene	5
1,2,4-Trichlorobenzene	5
1,1,1,2-Tetrachloroethane	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
1,2,3-Trichloropropane	5
1,2,4-Trimethylbenzene	5
1,3,5-Trimethylbenzene	5
Vinyl Acetate	10
Vinyl Chloride	10
o-Xylene	5
m,p-Xylene	5
Xylenes (total)	5

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A.: 8260 Low Conc. Water

Data File : C:\HPCHEM\1\DATA\9610219.B\1021904.D Vial: 9
Acq On : 21 Oct 96 12:24 pm Operator: COLLINS
Sample : VSTD005 IEA MSD9 Inst : MSD9
Misc : WATER LOW 1X Dilution: 1.00
Quant Time: Oct 21 14:18 1996

Method : C:\HPCHEM\1\DATA\9610219.B\8260LC.M
Title : 8260A IEA MSD9
Last Update : Mon Oct 21 16:06:54 1996
Response via : Multiple Level Calibration

Internal Standards	R.T.	QIon	Response	Conc Units	Dev(Min)
1) Pentafluorobenzene	9.16	168	1667300	5.00 µg/L	0.01
11) 1,4-Difluorobenzene	10.63	114	1873627	5.00 µg/L	0.00
37) Chlorobenzene-d5	17.06	117	1377997	5.00 µg/L	0.00
62) 1,4-Dichlorobenzene-d4	22.62	152	786567	5.00 µg/L	0.03
System Monitoring Compounds					**%Recovery**
14) 4-Bromofluorobenzene	19.87	95	937038	5.50 µg/L	110.08%
26) 1,2-Dichloroethane-d4	9.66	102	85673	5.81 µg/L	116.11%
35) Toluene-d8	13.77	98	1757279	5.50 µg/L	109.92%
Target Compounds					**Qvalue**
2) Acrylonitrile	5.92	53	23998	5.92 µg/L m	65
3) Allyl chloride	5.09	76	247066	5.64 µg/L	99
4) Dichlorodifluoromethane	2.20	85	469733	3.89 µg/L	98
5) 2,2-Dichloropropane	7.94	77	854728	5.48 µg/L	100
6) Iodomethane	4.64	142	1285768	5.91 µg/L	99
7) Methacrylonitrile	8.54	41	49927	5.27 µg/L #	87
8) Methyl-tert-Butyl ether	5.97	73	469765	6.80 µg/L	95
9) Trichlorofluoromethane	3.56	101	792725	4.98 µg/L	98
10) Vinyl acetate	7.00	43	321390	6.30 µg/L	99
12) Acetone	4.60	43	97609	32.28 µg/L m	98
13) Bromochloromethane	8.46	128	170524	5.76 µg/L	98
15) Bromomethane	3.01	94	424449	4.92 µg/L	99
16) 2-Butanone	8.17	43	186484	29.69 µg/L	100
17) Carbon disulfide	4.75	76	1375709	5.01 µg/L	99
18) Chloroethane	3.16	64	265489	6.00 µg/L	99
19) 2-Chloroethyl vinyl ether	12.99	63	123683	5.39 µg/L	96
20) Chloroform	8.67	83	1009394	5.79 µg/L	99
21) Chloromethane	2.46	50	356418	4.70 µg/L	99
22) Dibromomethane	11.84	93	229190	5.54 µg/L	95
23) 1,1-Dichloroethane	6.76	63	1006911	5.51 µg/L	99
24) 1,2-Dichloroethane	9.83	62	329524	5.94 µg/L	100
25) 1,1-Dichloroethene	4.40	96	511439	5.06 µg/L	99
27) cis-1,2-Dichloroethene	7.97	96	536091	5.64 µg/L	99
28) trans-1,2-Dichloroethene	5.89	96	581918	5.29 µg/L	99
29) 1,2-Dichloroethene (total)	7.97	96	536091	5.64 µg/L	99
30) 1,3-Dichloropropane	15.26	76	414188	5.57 µg/L	98
31) 1,1-Dichloropropene	9.33	75	789789	5.13 µg/L	99
32) Ethyl methacrylate	14.86	69	233930	5.40 µg/L	98
33) Methylene chloride	5.34	84	404274	5.73 µg/L	99
34) Methyl methacrylate	12.03	69	99522	4.91 µg/L	96
36) Vinyl chloride	2.59	62	410061	4.52 µg/L	99
38) Benzene	9.75	78	1590985	5.76 µg/L	100
39) Bromodichloromethane	12.22	83	800481	5.77 µg/L	99

PY 10/21/96

(#) = qualifier out of range (m) = manual integration

Data File : C:\HPCHEM\1\DATA\9610219.B\1021904.D Vial: 9
Acq On : 21 Oct 96 12:24 pm Operator: COLLINS
Sample : VSTD005 IEA MSD9 Inst : MSD9
Misc : WATER LOW 1X Dilution: 1.00
Quant Time: Oct 21 14:18 1996

Method : C:\HPCHEM\1\DATA\9610219.B\8260LC.M
Title : 8260A IEA MSD9
Last Update : Mon Oct 21 16:06:54 1996
Response via : Multiple Level Calibration

	Compound	R.T.	QIon	Response	Conc	Unit		Qvalue
40)	Carbon tetrachloride	9.31	117	896851	4.73	µg/L		100
41)	Chlorodibromomethane	15.75	129	540921	5.86	µg/L		99
42)	Chlorobenzene	17.13	112	1370641	5.60	µg/L		99
43)	1,2-Dibromoethane	16.00	107	323184	5.25	µg/L		97
44)	cis-1,4-Dichloro-2-butene	19.80	75	10676	4.98	µg/L	m	47
45)	trans-1,4-Dichloro-2-buten	20.49	53	22293	4.89	µg/L	m	1
46)	1,2-Dichloropropane	11.60	63	521721	5.88	µg/L		99
47)	cis-1,3-Dichloropropene	13.21	75	656180	5.74	µg/L		100
48)	trans-1,3-Dichloropropene	14.51	75	425720	5.53	µg/L		99
49)	Ethylbenzene	17.42	91	2360820	5.51	µg/L		99
50)	2-Hexanone	15.68	43	329509	28.30	µg/L		98
51)	4-Methyl-2-pentanone	13.69	43	649205	30.46	µg/L		83
52)	Styrene	18.67	104	1323769	5.58	µg/L		98
53)	1,1,1,2-Tetrachloroethane	17.33	131	597151	5.38	µg/L		100
54)	Tetrachloroethene	15.13	164	846793	5.39	µg/L		99
55)	Toluene	13.92	91	1925953	5.67	µg/L		98
56)	1,1,1-Trichloroethane	8.97	97	985930	5.03	µg/L		99
57)	1,1,2-Trichloroethane	14.89	97	287613	6.20	µg/L		98
58)	Trichloroethene	11.12	95	731315	5.40	µg/L		100
59)	m,p-Xylene	17.70	106	1984002	10.86	µg/L		97
60)	o-Xylene	18.62	106	913435	5.40	µg/L		97
61)	Xylene (total)	18.62	106	913435	5.40	µg/L		97
63)	Bromoform	19.06	173	292577	5.30	µg/L		99
64)	Bromobenzene	20.17	156	612210	5.06	µg/L		99
65)	n-Butylbenzene	23.61	91	2135035	4.84	µg/L		96
66)	tert-Butylbenzene	21.68	119	2586701	5.10	µg/L		98
67)	sec-Butylbenzene	22.22	105	3119583	4.89	µg/L		100
68)	n-Propylbenzene	20.49	91	2621685	4.70	µg/L		99
69)	2-Chlorotoluene	20.63	91	1903334	5.51	µg/L		97
70)	4-Chlorotoluene	20.93	91	1786174	5.18	µg/L		91
71)	1,2-Dibromo-3-chloropropan	25.55	75	50882	5.51	µg/L	m	3
72)	1,2-Dichlorobenzene	23.56	146	929611	5.29	µg/L		97
73)	1,3-Dichlorobenzene	22.45	146	1117091	5.07	µg/L		99
74)	1,4-Dichlorobenzene	22.67	146	1259749	5.56	µg/L		98
75)	Hexachlorobutadiene	26.94	225	784583	4.49	µg/L		100
76)	p-Isopropyltoluene	22.61	119	2400881	4.94	µg/L		99
77)	Isopropylbenzene	19.51	105	2495247	5.03	µg/L		99
78)	Naphthalene	27.11	128	566700	4.70	µg/L		100
79)	Pentachloroethane	21.68	167	366270	4.81	µg/L		99
80)	1,1,2,2-Tetrachloroethane	20.27	83	301923	5.81	µg/L		98
81)	1,2,4-Trichlorobenzene	26.72	180	698902	5.25	µg/L		92
82)	1,2,3-Trichlorobenzene	27.41	180	617726	5.58	µg/L		92
83)	1,2,3-Trichloropropane	20.33	75	206270	5.24	µg/L	#	11
84)	1,3,5-Trimethylbenzene	20.94	105	1796895	5.27	µg/L		100

(#) = qualifier out of range (m) = manual integration

Data File : C:\HPCHEM\1\DATA\9610219.B\1021904.D Vial: 9
Acq On : 21 Oct 96 12:24 pm Operator: COLLINS
Sample : VSTD005 IEA MSD9 Inst : MSD9
Misc : WATER LOW 1X Dilution: 1.00
Quant Time: Oct 21 14:18 1996

Method : C:\HPCHEM\1\DATA\9610219.B\8260LC.M
Title : 8260A IEA MSD9
Last Update : Mon Oct 21 16:06:54 1996
Response via : Multiple Level Calibration

Compound	R.T.	QIon	Response	Conc Unit	Qvalue
85) 1,2,4-Trimethylbenzene	21.82	105	1747553	5.22 µg/L	99

(#) = qualifier out of range (m) = manual integration

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Quantitation Report

Data File : C:\HPCHEM\1\DATA\9611014.B\1101403A.D Vial: 2
Acq On : 1 Nov 96 12:35 pm Operator: WESTHEAD
Sample : SSTD0504Z SVCLP534-H Inst : MSD4
Misc : WATER LOW 1X IEA MSD4 Multiplr: 1.00
Quant Time: Nov 1 13:13 1996

Method : C:\HPCHEM\1\DATA\9611014.B\IEA8270.M
Title :
Last Update : Thu Oct 31 10:08:49 1996
Response via : Initial Calibration

Internal Standards	R.T.	QIon	Response	Conc Units	Dev(Min)
1) 1,4-dichlorobenzene-d4	9.67	152	610069	40.00	0.00
20) Naphthalene-d8	12.57	136	2484033	40.00	-0.01
35) Acenaphthene-d10	16.77	164	1408244	40.00	-0.02
56) Phenanthrene-d10	20.29	188	2268873	40.00	-0.02
68) Chrysene-d12	26.69	240	2046152	40.00	-0.03
77) Perylene-d12	29.95	264	2133040	40.00	-0.05
System Monitoring Compounds					%Recovery
4) 2-Fluorophenol	7.11	112	1084617	45.43	30.285%
5) Phenol-d5	9.13	99	1548055	46.33	30.884%
9) 2-Chlorophenol-d4	9.27	132	1055047	45.69	30.458%
13) 1,2-Dichlorobenzene-d4	10.09	152	678701	51.03	51.031%
21) Nitrobenzene-d5	11.00	82	1320393	45.56	45.556%
39) 2-Fluorobiphenyl	15.21	172	1996378	46.40	46.404%
55) 2,4,6-Tribromophenol	18.71	330	376277	47.38	31.586%
71) Terphenyl-d14	24.20	244	1977423	43.89	43.889%
Target Compounds					Qvalue
2) Pyridine	4.21	79	1083823	38.20	88
3) N-Nitrosodimethylamine	4.26	74	757579	41.83	98
6) Phenol	9.15	94	1540745	45.45	98
7) Bis(2-Chloroethyl)ether	9.22	93	1171113	45.46	99
8) 2-Chlorophenol	9.31	128	1061948	45.17	100
10) 1,3-Dichlorobenzene	9.58	146	1165455	49.47	99
11) 1,4-Dichlorobenzene	9.71	146	1196934	49.64	98
12) 1,2-Dichlorobenzene	10.12	146	1110125	50.93	98
14) Benzyl Alcohol	10.10	108	809337	47.67	98
15) 2,2'-oxybis(1-Chloropropan	10.41	45	1165623	32.68	99
16) 2-Methylphenol	10.43	108	1086421	46.71	99
17) N-Nitroso-di-n-propylamine	10.74	70	924364	43.19	98
18) Hexachloroethane	10.78	117	498041	47.63	97
19) 4-Methylphenol	10.77	108	1188129	47.67	99
22) Nitrobenzene	11.04	77	1308046	44.34	99
23) Isophorone	11.57	82	2623753	44.12	99
24) 2-Nitrophenol	11.77	139	670482	50.17	99
25) 2,4-Dimethylphenol	11.96	107	1169690	47.24	100
26) bis(2-Chloroethoxy)methane	12.14	93	1505576	46.92	100
27) 2,4-Dichlorophenol	12.34	162	900859	45.94	98
28) Benzoic acid	12.35	122	654871	36.77	98
29) 1,2,4-Trichlorobenzene	12.49	180	839393	41.07	99
30) Naphthalene	12.62	128	2926617	48.50	100
31) 4-Chloroaniline	12.86	127	1341545	47.13	100
32) Hexachlorobutadiene	13.06	225	491431	44.10	99
33) 4-Chloro-3-methylphenol	14.09	107	1196814	51.79	98

(#) = qualifier out of range (m) = manual integration

Quantitation Report

Data File : C:\HPCHEM\1\DATA\9611014.B\1101403A.D Vial: 2
Acq On : 1 Nov 96 12:35 pm Operator: WESTHEAD
Sample : SSTD0504Z SVCLP534-H Inst : MSD4
Misc : WATER LOW 1X IEA MSD4 Multiplr: 1.00
Quant Time: Nov 1 13:13 1996

Method : C:\HPCHEM\1\DATA\9611014.B\IEA8270.M
Title :
Last Update : Thu Oct 31 10:08:49 1996
Response via : Initial Calibration

Compound	R.T.	QIon	Response	Conc Unit	Qvalue
34) 2-Methylnaphthalene	14.26	142	2141543	52.32	99
36) Hexachlorocyclopentadiene	14.79	237	531781	43.23	100
37) 2,4,6-Trichlorophenol	15.04	196	597717	40.26	99
38) 2,4,5-Trichlorophenol	15.14	196	646237	40.21	98
40) 2-Chloronaphthalene	15.41	162	1906895	48.99	99
41) 2-Nitroaniline	15.79	65	861322	46.42	99
42) Dimethylphthalate	16.32	163	2486105	48.42	100
43) Acenaphthylene	16.41	152	3386673	55.41	99
44) 2,6-Dinitrotoluene	16.48	165	594346	46.74	99
45) 3-Nitroaniline	16.80	138	719647	50.64	96
46) Acenaphthene	16.85	153	2031766	52.49	99
47) 2,4-Dinitrophenol	17.05	184	358041	42.18	95
48) Dibenzofuran	17.24	168	2638311	46.19	99
49) 4-Nitrophenol	17.33	109	543819	56.41	98
50) 2,4-Dinitrotoluene	17.41	165	829156	44.19	99
51) Diethylphthalate	18.02	149	2903518	53.48	100
52) Fluorene	18.06	166	2160222	48.30	98
53) 4-Chlorophenyl-phenylether	18.09	204	911536	44.24	99
54) 4-Nitroaniline	18.30	138	763544	49.74	99
57) 4,6-Dinitro-2-Methylphenol	18.39	198	470312	48.81	93
58) N-Nitrosodiphenylamine (1)	18.43	169	1515148	56.12	99
59) Azobenzene	18.47	77	3455505	58.37	99
60) 4-Bromophenyl-phenylether	19.25	248	576548	54.38	98
61) Hexachlorobenzene	19.58	284	664299	50.96	99
62) Pentachlorophenol	20.05	266	412957	42.14	98
63) Phenanthrene	20.34	178	2802932	51.73	99
64) Anthracene	20.45	178	2831371	51.93	100
65) Carbazole	20.91	167	3066638	61.00	100
66) Di-n-butylphthalate	21.93	149	5163171	67.74	100
67) Fluoranthene	23.21	202	3034478	51.12	99
69) Benzidine	23.62	184	1731705	97.83	100
70) Pyrene	23.74	202	3146787	43.04	99
72) Butylbenzylphthalate	25.51	149	2455192	54.89	100
73) Benzo(a)anthracene	26.64	228	3014183	45.78	100
74) 3,3'-Dichlorobenzidine	26.69	252	1221206	55.28	100
75) Chrysene	26.75	228	2868051	45.64	99
76) bis(2-Ethylhexyl)phthalate	26.96	149	3414509	58.08	100
78) Di-n-octylphthalate	28.36	149	5813566	62.63	100
79) Benzo(b)fluoranthene	29.09	252	3207817	46.72	98
80) Benzo(k)fluoranthene	29.15	252	2771607	51.21	98
81) Benzo(a)pyrene	29.81	252	2873327	49.10	100
82) Indeno(1,2,3-cd)pyrene	32.92	276	3170079	46.74	99
83) Dibenz(a,h)anthracene	32.98	278	2522028	50.13	99
84) Benzo(g,h,i)perylene	33.80	276	2690419	47.35	99

(#) = qualifier out of range (m) = manual integration

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1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director _____

Quality Assurance Manager _____ DRAFT

2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in multimedia, multi-concentration samples.

2.2 Refer to Table 6 of this SOP for parameters.

2.3 The document control number for this SOP is GCSDRAFT.NC.

3.0 SUMMARY OF METHOD

3.1 This method outlines the gas chromatographic procedure for the detection of organochlorine pesticides and polychlorinated biphenyls. Samples are extracted using the proper extraction technique. The extracts are analyzed by gas chromatography with an electron capture detector.

3.2 This SOP is based on the following methods:

- EPA Method 8081 (Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique)
- EPA Method 8000A (Gas Chromatography)

4.0 INTERFERENCES

4.1 Phthalate esters can interfere with pesticide determination; avoid any contact with plastic to best minimize this problem.

4.2 Sulfur is also an interference; this can be removed by performing sulfur clean-up on the extract using TBA-Sulfite. Refer to the SOP for Sulfur Removal, IEA Doc# GCS01500.NC.

4.3 If an extract is to be analyzed for PCB's only, then an optional sulfuric acid cleanup may be performed. This cleanup procedure will remove contamination which may interfere with the analysis of the eluting PCB's, however, the procedure will also cause

degradation of other pesticide and surrogate compounds. If contamination, due to sample matrix, is suspected in the sample, or the original extract analysis exhibits interferences, then the extract may be cleaned up with concentrated sulfuric acid and reanalyzed. A portion of the original method blank is also acid cleaned and analyzed.

4.3.1 Acid cleanup is performed by adding one part sulfuric acid to two parts of the hexane extract in a clean vial. Tighten the vial and agitate the sample for thirty seconds. After cleanup, pipet the extract layer to another vial. Repeat the cleanup procedure until the acid layer is light brown to orange in color. Complete the acid cleanup procedure by performing a sulfur cleanup. This cleanup must also be performed on the corresponding method blank and QC if all samples required cleanup. Refer to the SOP for Sulfur Removal, IEA Doc# GCS01500.NC. Document in the case narrative each sample on which acid and/or sulfur cleanup was performed.

4.3.2 Florisil cleanup is an optional clean procedure used as necessary to eliminate interfering compounds. Refer to USEPA CLP OLMO3.1 Statement of Work, pg. D-53/Pest, Section 10.1.8.2 on Florisil cleanup using Florisil Cartridges.

5.0 SAFETY

5.1 All compounds listed in Tables 1-7 (see attached documents) along with their respective solvents (i.e., hexane, isooctane, acetone, toluene) are toxic. Non-powdered polyvinyl gloves and safety glasses should be worn at all times when handling any of these solvents and compounds. Also, all work with these chemicals should be performed in a fume hood.

5.2 Material Safety Data Sheets (MSDS) for all chemicals used in operations are present in the laboratory for immediate access. Specific MSDS's should be reviewed for a complete knowledge of the toxicity and precautions of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

6.1.1 Soil samples are collected with 250 ml or 500 ml amber glass containers with Teflon-coated liner.

6.1.2 Water sample are collected in 2x1 liter amber glass containers with Teflon-lined lids.

6.1.3 Precleaned certified sample bottles are purchased from a commercial vendor.

6.1.4 Sample bottles are never cleaned and reused.

6.2 Sample Collection

6.2.1 Samples are collected as grab samples in the field.

6.2.2 A cooler with either ice or blue ice should be available at the sampling site.

6.2.3 Samples are secured against breakage in the coolers and kept at 4 deg. C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

6.3 Sample Preservation

6.3.1 Samples are preserved by cooling to 4 deg. C.

6.4 Holding Times

6.4.1 Soil samples must be extracted within 14 days from sampling.

6.4.2 Water samples must be extracted within 7 days from collection.

6.4.3 Extracts must be analyzed within 40 days of extraction.

7.0 APPARATUS AND MATERIALS

7.1 Sample extracts are analyzed on a gas chromatograph (GC) equipped with an electron-capture detector (ECD), autosampler, data collection system and all other required accessories. The following columns are acceptable for quantitation and confirmation:

7.1.1 RTX-35 30 meter, 0.53mm ID 0.83um film thickness, Restek Corporation Cat #10455

7.1.2 DB-1701 30 meter, 0.53mm ID 1.5um film thickness, J&W Scientific CAT #125-0732

7.2 Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs

7.3 0.8 ml and 1.8 ml autosampler crimptop vials and caps.

7.4 Vial crimper

7.5 Borosilicate glass transfer pipets/transfer bulbs.

7.6 Safety glasses, non-powdered polyvinyl gloves, fume hood.

7.7 Properly cooled refrigerators separately for sample and standard storage.

7.8 Standard and Instrument Maintenance logbooks.

7.9 Sample extract refrigerator log-in books.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Solvents: All solvents should be pesticide grade or equivalent.

8.1.1 Hexane

8.1.2 Acetone

8.1.3 Toluene

8.1.4 Isooctane (2,2,4-trimethylpentane)

8.2 Standards

8.2.1 Calibration Stock Standard is prepared by diluting 1.0 ml of NSI Custom Pesticide Mixture Q-1132-01 to 100 mL in a pre-rinsed wolumetric flask with iso-octane. See Table 1 for final concentrations of compounds.

8.2.2 Single-component Calibration Standards at a minimum of 5 concentration levels are prepared through dilution of the calibration stock standard. The concentration should correspond to the expected range of concentrations found in actual samples and should bracket the linear range of the instrument. See Table 1 for final component concentrations.

8.2.3 Multicomponent Standards - A five point calibration curve is generated for multicomponent pesticide compounds Chlordane (technical) and Toxaphene when they are detected in a sample. Singel-point standards are analyzed after the single component pesticide calibration curves for identification purposes. For Multi-component Stock and Standard concentrations see Tables 2.0 & 2.1.

8.2.4 Surrogate Standard is prepared to monitor the performance of the extraction and analytical system. Two surrogates, 2,4,5,6-tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), are spiked into each sample, blank and QC sample prior to extraction. (See Table 3 for spike concentrations).

8.2.5 Laboratory Control Sample (LCS) and Matrix Spiking Stock Standard Solution is prepared by diluting 1.0 ml of NSI Pesticide Mix Q-1065A-01 to 50 mL in a pre-rinsed volumetric flask with a 90% Iso-octane/10% Acetone mix. The working spike solution prepared for extractions is made by diluting 10 mL of the above stock solution to 100 mL in a pre-rinsed volumetric flas with an 80% Methanol/20% Aceton mix. 1.0 mL of the working solutioin is spiked into each aliquot of the sample for the matrix spike and matrix spike duplicate, and to 1.0 L of reagent water for the LCS sample. (See Table 4.)

8.2.5.1 If the batch of samples is for PCB only analysis, an MS/MSD of Aroclors only, is used. (See Table 4.1).

8.2.6 Instrument Breakdown Standard is prepared as a solution of p,p'DDT and Endrin at a concentration near the midlevel concentration of the calibration standards. (See Table 5).

8.3 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer.

8.4 All stock and working standards are stored in amber screw top bottles at 4 deg. C and replaced after 6 months or earlier if necessary.

8.5 All standard preparation must be documented in the Standards Logbook (Attachment I).

9.0 CALIBRATION

9.1 Retention Time (RT) windows are established by making three injections of the mid concentration standards throughout the course of a 72-hour period and calculating 3 x $\pm$ the standard deviation. Retention time windows shall be calculated for each compound on each GC column whenever a new column is installed.

9.1.1 RT windows are established as described above, however, the laboratory has established minimum RT windows as found in Table 8.

9.2 Calibration Standards

9.2.1 Initial calibration standards are analyzed by injecting 1 uL of each of the five levels of the single component calibration standard, and one level of all other multi-response pesticides/PCB's.

9.2.1.1 When analyzing for Aroclors only or when an Aroclor or multi-component pesticide is detected, an initial calibration consisting of five standard concentration levels is injected and the sample is re-analyzed against this five-point calibration curve. See Table 2.1 for Aroclor and multi-component pesticide curve standard concentrations.

9.2.2 The working calibration range of this method is defined by the initial calibration curve. All extracts with target compounds exceeding the curve must be diluted to within the calibration range.

9.3 The daily calibration verification is acceptable if all of the following are true.

9.3.1.1 The breakdown of Endrin or p'p'DDT is <= 15% based on the presence of Endrin Aldehyde and Endrin Ketone for Endrin, p,p'DDD and p,p'DDE for

p,p'DDT. If the breakdown does exceed 15% then corrective action must be taken prior to continuing the calibration verification.

$$\% \text{ breakdown for p'p'DDT} = \frac{\text{DDD peak area + DDE peak area}}{\text{Total DDT peak area (DDT+DDD+DDE)}} \times 100$$

% breakdown for Endrin =

$$\frac{\text{Endrin aldehyde peak area + Endrin Ketone peak area}}{\text{Total endrin peak area (Endrin+Endrin aldehyde+Endrin Ketone)}} \times 100$$

9.3.1.2 The midlevel calibration standard must have all compounds (+/-) 15% of their expected value. Because of the low concentration of pesticide standards injected on a GC/EC, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a pesticide standard mixture approximately 20 times more concentrated than the midlevel standard.

If the midlevel calibration standard fails, then it can be rerun. A new calibration curve will be run, if warranted, due to repeated failure.

9.3.1.3 An instrument blank standard is run after the midlevel calibration standard and prior to any samples to ensure the instrument and its autosampler are clean.

9.4 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range. Multicomponent targets must also be diluted so that the largest peak in the multicomponent does not exceed the response of the largest peak in the high level single-component calibration standard.

9.5 Calibration Verification (Calibration Check Standard - CCS)

9.5.1 A single midlevel standard (standard mix or multi-compound), is analyzed at a minimum of every 10 samples and at the end of the analysis sequence. The calibration factor for each compound to be quantitated must not exceed a 15% difference when compared to the average calibration factor from the calibration curve. When this criteria is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is required before recalibrating. All samples that were injected prior to the standard exceeding the criteria must be reinjected if the initial analysis indicated the presence of the specific target analytes that exceeded the criteria.

9.6 Calculations for water samples:

9.6.1 Quantitation of single-component target analytes is as follows:

$$ug/L = \frac{\text{Area of sample peak X Final volume of extract (uL) X Dilution Factor}}{\text{Avg Calibration Factor of Standard X Sample Volume extracted (mLs)}}$$

$$CF = \frac{\text{Peak area of the Standard}}{\text{Mass injected (ng)}}$$

9.6.2 An average calibration factor is calculated for each compound and surrogate from the initial calibration. Multi-component analytes are calculated by adding the areas of three to five of the largest peaks common to the sample and the standard and using this combined area in the following calculation:

$$ug/L = \frac{\text{(X)Final Volume of extract (uL) X Dilution Factor}}{\text{(Y)Sample volume extracted (mLs)}}$$

where:

X = the combined area of 3-5 peaks from sample.

Y = the average CF of the combined areas of 3-5 peaks from initial calibration standards.

9.7 Calculations for soil and oil samples:

9.7.1 The calculation for quantitation for single component target analytes is as follows:

$$ug/kg = \frac{\text{Area of sample peak X Final Volume of Extract (uL) X Dilution Factor}}{\text{Average CF of Standard X Sample volume extracted (g) X Decimal \% Solids}}$$

$$CF = \frac{\text{Peak Area of the Standard}}{\text{Mass injected (ng)}}$$

9.7.2 Multicomponent analytes are calculated by adding three to five of the largest peaks common to the sample and the standard, and using this combined area in the following calculation:

$$ug/kg = \frac{\text{(X)Final Volume of extract (uL) X Dilution Factor}}{\text{(Y)Sample volume extracted (g) X Decimal \% solids}}$$

where:

X = the combined area of 3-5 peaks from sample.

Y = the average CF of the combined areas of 3-5 peaks from initial calibration standards.

Note: Oils will not have a percent solids.

9.7.3 When more than one multicomponent is detected in a sample, non-overlapping peaks are chosen for quantitation. If it is not possible to choose non-overlapping peaks, peaks with the least amount of overlap are chosen.

9.8 Corrective Action for Calibration

9.8.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

9.9 Corrective Action for Calibration Verification

9.9.1 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective actions to achieve the criteria.

9.9.2 Major corrective actions such as replacing the column or detector will require a new initial calibration.

9.9.3 Minor corrective actions may not require a new initial calibration provided a calibration check meets all acceptance criteria.

10.0 QUALITY CONTROL

10.1 Refer to Table 6 for Practical Quantitation Limits (PQL's) for all compounds.

10.2 Matrix spike (MS), matrix spike duplicates (MSDs) and Laboratory Control Samples (LCS) are extracted within every batch of 20 samples or less. Recoveries must be within the limits listed in Table 7 of this SOP. If these criteria are not met, but the LCS data meet all of the recovery criteria, then the MS/MSD are documented as exhibiting suspected matrix interferences. If the LCS fails, check for instrument and/or column related problems and reanalyze the LCS. If the problem is corrected, the samples are reanalyzed. If these LCS still fails, the sample batch is reextracted and reanalyzed for an LCS compounds with failing recoveries below the lower acceptance range. If target compounds of interest have a recovery exceeding the highest acceptance range and the samples have no detected results for these target compounds, the data is reported with

a discussion in the project narrative. In this instance, no quantitation limits or reported target compounds concentrations are affected.

10.3 Blanks

10.3.1 Frequency - method blanks must be analyzed with each batch of 20 samples of a similar matrix, or whenever samples are extracted by the same procedure, whichever is more frequent.

10.3.2 Method blanks are spiked with surrogates,extracted and analyzed following the same procedure that is used with the associated samples. A water method blank is one liter of reagent water and a soil method blank is 30 g of sodium sulfate.

10.3.3 Method blanks must contain less than the PQL for all the target compounds listed in Table 1.0.

10.3.4 All samples associated with an unacceptable method blank must be reextracted and reanalyzed.

10.4 Surrogates

10.4.1 The surrogates TCX and DCB are added to each sample, standard, blank and QC prior to extraction.

10.4.2 The advisory QC limits for surrogate recovery are listed in Table 3.1 and pertain to all samples, blanks and spikes. These limits are only advisory; therefore no further action is required if the criteria limits are not achieved

10.5	Calibration check sample	One per 24 hours and after every 10 samples
	Breakdown standard	One per 24 hours
	Instrument blank	One per 24 hours
	MS/MSD	One pair every 20 samples
	LCS	Every extraction batch (20 samples or less)
	Surrogates	Every sample and standard and blank
	Method blank (extracted)	Every extraction batch (20 samples or less)

10.6 Chain of Custody Record

10.6.1 All sampels are requested from the Sample Custodia using the Sample Request /Delivery Form (Attachment II).

10.6.2 When samples designated for Level III reports (or if specified by the client) are removed from storage for preparation or analysis they must be signed out utilizing the IEA internal chain of custody (COC). The samples shall then be signed back

in on the internal COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation for analysis.

10.7 MDLs are determined in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Two HP5890 Series II Gas Chromatographs (GCs) with dual EC's and 7673A Twin Tower Autosampler is currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and conditions are as follows:

a)

Front Injection port:	30 meter RTS-35 (See Section 7.1)
Column flow:	4.0 ml/min He
Detector Make-up Flow:	55 ml/min P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C

Temperature Programming:

Initial Temperature:	160 deg. C Hold 1.0 min.
Initial Ramp:	3 degrees/min to 200 deg. C, Hold 3.0 min.
Final Ramp and Temp.:	15 degrees/min to 270 deg. C, Hold 16 min.

b)

Back Injection Port	DB-1701 (See Section 7.1)
Column flow:	5 ml/min He
Detector Make-up Flow:	65 ml/min P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C

Temperature Programming:

Initial Temperature:	160 deg. C, Hold 1.0 min.
Initial Ramp:	3 degrees/min to 200 deg. C, Hold 3.0 min.
Final Ramp and Temp.:	15 degrees/min to 270 deg. C, Hold 16 min.

11.2 Soil samples are prepared in accordance with the SOP for SW-846 Method 3550 (Pesticide's/PCB's in Soil), IEA Doc# SPS01500.NC.

11.3 Water samples are prepared in accordance with the SOP for SW-846 Method 3510 (Pesticide's/PCB's in Water), IEA Doc# SPS01600.NC.

11.4 Sample Analysis

11.4.1 1.0 uL of extract is injected onto the GC column by an autosampler and the temperature program sequence is stared.

11.4.2 If a dilution is employed to bring a peak within the calibration range, both results are reported. The resulting change in quantitation limits and surrogate recovery must be reported for the diluted samples.

11.5 Qualitative Analysis

11.5.1 Target Compounds

11.5.1.1 The identification of a single component pesticide is based on the retention time data. The RT of a peak can be verified only from an on-scale chromatogram.

11.5.1.2 If a compound falls within the RT windows of the compound in the calibration curve, and is greater than the PQL, that sample would require confirmation by analysis on a second column.

11.6 Quantitative Analysis

11.6.1 Target Compounds

11.6.1.1 Target compounds are quantitated by the external standard technique using the peak area and the calibration factor determined during the initial calibration sequence.

11.6.1.2 When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

11.6.1.3 When a compound exceeds the linear working range of the initial calibration, the sample must be diluted to bring the analyte to within the calibration range.

11.7 Data Acquisition

11.7.1 Data Acquisition and System Operation

11.7.1.1 Data is acquired from sample analyses using the Hewlett-Packard 1000 computer system with RTE operating system and LDS processing software. Analytical batches are set up using LDS with all the associated sample ID, dilution, and data file information. The analytical sequence setup mirrors the autosampler sequence setup. Automated post-acquisition quantitation is queued with the appropriate method file. The sequence is assigned using the LDS program and AS and starts acquiring when the A/D converter gets a start signal from the GC.

11.8 Manual integration Editing Flags

11.8.1 Manual integration shall be performed when the automated integration does not yield correctly u=integrated baselines. Manual integrations re flagged by the data system with the "FF" qualifier beside any manually integrated area. The analyst shall initial and date the hardcopy report for all manual integration performed.

12.0 CALCULATIONS

12.1 Calculation of Calibration Factor

$$CF = \frac{\text{Peak Area of the Standard}}{\text{Mass injected (ng)}}$$

12.2 Calculation of % Difference:

$$\% \text{ Difference} = \frac{|\text{Calculated conc. - expected conc.}|}{\text{Expected conc.}} \times 100$$

12.3 Calculation of spike and QC check recoveries:

$$\% \text{ Recoveries} = \frac{\text{Amount recovered}}{\text{Amount added}} \times 100\%$$

12.4 Calculation of Percent Moisture

$$\% \text{ Moisture} = \frac{\text{Wet Sample (g) - Dry Sample (g)}}{\text{Wet Sample (g)}} \times 100$$

12.5 Calculation of Adjusted Practical Quantitation Limit

$$\text{Adjusted CRQL} = \frac{(\text{CRQL}) \times D_f}{D}$$

Where:

$$D = \frac{100 - \% \text{ Moisture}}{100}$$

D_f = Dilution factor

12.6 Calculation for Target Compounds

See Sections 9.6 and 9.7.

13.0 ACCEPTANCE OF DATA

13.1 A calibration check standard is analyzed to verify the calibration curve by analysis of the midlevel single-component calibration standard is obtained if the calculated concentration of all the compounds is (+/-) 15% of the expected value. The Calibration Check Standard is analyzed after every 10 samples.

13.2 Instrument Breakdown of DDT and Endrin is considered under control if the % Breakdown of each analyte is <= 15%.

13.3 The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.4 Method blanks are extracted with every batch of up to 20 samples or less to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.5 Matrix Spikes and Matrix Spike Duplicates are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed in Table 7.

13.6 Laboratory Control Samples are extracted with every batch of 20 samples. Acceptance criteria are listed in Table 7.

14.0 REPORTING OF RESULTS

14.1 All samples will have their target analytes reported in ug/L for waters and ug/Kg for soils. For an analyte to be reported, the concentration must be above the MDL. Results above the MDL and below the PQL will be flagged "J" as estimated, if requested by the client.

14.2 All results will be reported to two significant figures. Figures are rounded when the final concentration is achieved.

14.3 If it is determined that analytes are not present in the sample, the analytes will be reported as BQL (below quantitation limit).

14.4 The Quantitation Limit will be elevated proportionately in samples requiring dilutions.

14.5 Three different report levels are available:

14.5.1 A Level I data package includes: sample results, blank results and sampling COCs.

14.5.2 A Level II data package includes: Sample results, blank results, matrix spike results, matrix spike duplicate summary data, surrogate recovery data and sampling COCs.

14.5.3 A Level III data package includes: Sample results, blank results, matrix spike results, matrix spike duplicate summary data, project narrative, LCS results, surrogate recovery data, IEA assigned # (cross reference) index and sampling and internal COCs.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Sulfur Removal, IEA Doc# GCS01500.NC.

15.2 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.3 SOP for SW-846 Method 3550 (Pesticide's/PCB's in Soil), IEA Doc# SPS01500.NC.

15.4 SOP for SW-846 Method 3510 (Pesticide's/PCB's in Water), IEA Doc# SPS01600.NC.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, September 1994 Revision, USEPA, Method 8081.

16.2 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, September 1994 Revision, USEPA, Method 8000.

16.3 USEPA CLP OLMO3.1 Statement of Work.

17.0 SUBSTANTIVE REVISIONS

17.1 Draft Issue (12/10/96).

TABLE 1 - Single Component Calibration Concentrations, ug/mL

Analyte	Stock	Cal A	Cal B	Cal C	Cal D	Cal E
Aldrin	1.0	0.005	0.01	0.025	0.05	0.10
Dieldrin	2.0	0.01	0.02	0.05	0.10	0.20
p,p'DDT	6.0	0.03	0.06	0.15	0.30	0.60
p,p'DDE	2.0	0.01	0.02	0.05	0.10	0.20
p,p'DDD	6.0	0.03	0.06	0.15	0.30	0.60
Endosulfan I	2.0	0.01	0.02	0.05	0.10	0.20
Endosulfan II	2.0	0.01	0.02	0.05	0.10	0.20
Endosulfan Sulfate	6.0	0.03	0.06	0.15	0.30	0.60
Endrin	2.0	0.01	0.02	0.05	0.10	0.20
Endrin Aldehyde	6.0	0.03	0.06	0.15	0.30	0.60
Heptachlor	1.0	0.005	0.01	0.025	0.05	0.10
Heptachlor Epoxide	1.0	0.005	0.01	0.025	0.05	0.10
alpha-BHC	1.0	0.005	0.01	0.025	0.05	0.10
beta-BHC	1.0	0.005	0.01	0.025	0.05	0.10
gamma-BHC	1.0	0.005	0.01	0.025	0.05	0.10
delta-BHC	1.0	0.005	0.01	0.025	0.05	0.10
Methoxychlor	6.0	0.03	0.06	0.15	0.30	0.60
Endrin Ketone	6.0	0.03	0.06	0.15	0.30	0.60
TCX	1.0	0.005	0.01	0.025	0.05	0.10
DCB	1.0	0.005	0.01	0.025	0.05	0.10

TABLE 2 - Multi-Component Concentration, ug/mL

Multi-Component	Stock	Working Standard
AR 1221	10	0.20
AR 1232	10	0.10
AR 1242	10	0.10
AR 1248	10	0.10
AR 1254	10	0.10
Technical Chlordane	50.0	0.20
Toxaphene	10.0	0.50

TABLE 2.1
Multi-Component Concentration for
5-Point Calibrations, ug/mL

Compound	Stock	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
AR1221	10	0.25	0.50	1.0	2.0	4.0
AR1232	10	0.25	0.50	1.0	2.0	4.0
AR1242	10	0.25	0.50	1.0	2.0	4.0
AR1248	10	0.25	0.50	1.0	2.0	4.0
AR1254	10	0.25	0.50	1.0	2.0	4.0
AR1660	10	0.25	0.50	1.0	2.0	4.0
Toxaphene	10	0.25	0.50	1.0	1.5	2.0
Chlordane (Technical)	50	0.10	0.20	0.5	1.0	2.0

TABLE 3 - Surrogate Mix, ug/mL

Surrogate	Stock	Mix for Extractions (added to each sample and spike)
TCX	2.0	0.20
DCB	2.0	0.20

Table 3.1
Surrogate Recovery Limits

Surrogate	Soil	Water
TCX	60-150	60-150
DCB	60-150	60-150

TABLE 4 - QC Check and Matrix Spike, ug/mL

Analyte	Stock	Standard for Extractions
Aldrin	1.0	0.20
Dieldrin	1.0	0.20
p,p'DDT	5.0	1.0
p,p'DDE	1.0	0.2
p,p'DDD	5.0	1.0
Endosulfan I	1.0	0.20
Endosulfan II	5.0	1.0
Endosulfan Sulfate	5.0	1.0
Endrin	5.0	1.0
Endrin Aldehyde	5.0	1.0
Heptachlor	1.0	0.20
Heptachlor Epoxide	1.0	0.20
alpha-BHC	1.0	0.20
beta-BHC	1.0	0.20
gamma-BHC	1.0	0.20
delta-BHC	1.0	0.20
Methoxychlor	10.0	2.0
Endrin Ketone	5.0	1.0

TABLE 4.1
Aroclor Matrix Spike Solution (ug/mL)

Compound	Stock	Std. Mix for Extractions (Added to each Aroclor MS)	Water Control Limits	Soil Control Limits
AR-1260	1000	10	8-127	8-127

TABLE 5 - Breakdown Std Conc., ug/mL

Compound	Stock (individual compound)	Standard (working)
Endrin	1.0	0.1
p,p'DDT	1.0	0.1

TABLE 6 - Practical Quantitation Limits (PQL)

Analyte	PQLs for Waters (ug/mL)	PQLs for Soils (ug/kg)
alpha-BHC	0.05	8.0
beta-BHC	0.05	8.0
delta-BHC	0.05	8.0
gamma-BHC (Lindane)	0.05	8.0
Heptachlor	0.05	8.0
Aldrin	0.05	8.0
Heptachlor Epoxide	0.05	8.0
Endosulfan I	0.05	8.0
Dieldrin	0.10	16
p,p'DDE	0.10	16
Endrin	0.10	16
Endosulfan II	0.10	16
p,p'DDD	0.10	16
Endosulfar Sulfate	0.10	16
p,p'DDT	0.10	16
Methoxychlor	0.50	80
Toxaphene	1.0	160
AR 1016	1.0	80
AR 1221	2.0	80
AR 1232	1.0	80
AR 1242	1.0	80
AR 1248	1.0	80
AR 1254	1.0	160
AR 1260	1.0	160
Tech. Chlordane	0.20	80
Endrin Aldehyde	0.10	8.0
Endrin Ketone	0.10	8.0

Table 7
Matrix Spike
& Laboratory Control Sample Acceptance Criteria

Compound	Matrix Spike Recovery Limits (Soils)	Matrix Spike Recovery Limits (Waters)	LCS Recovery Limits (Soils)	LCS Recovery Limits (Waters)
alpha-BHC	37-134	37-134	43-105	41-126
gamma-BHC	32-127	32-127	52-108	60-127
Heptachlor	34-111	34-111	52-111	61-111
Aldrin	42-122	42-122	42-122	64-113
beta-BHC	17-147	17-147	63-125	17-147
delta-BHC	19-140	19-140	19-140	19-140
Heptachlor epoxide	37-142	37-142	54-121	76-128
Endosulfan I	45-153	45-153	66-138	87-143
4,4'-DDE	30-145	30-145	61-143	46-145
Dieldrin	36-146	36-146	57-146	75-146
Endrin	30-147	30-147	55-126	58-133
4,4"-DDD	31-141	31-141	53-127	69-141
Endosulfan II	10-200	10-200	47-121	53-153
4,4'-DDT	25-160	25-160	25-160	46-134
Endrin Aldehyde	10-200	10-200	10-200	10-200
Methoxychlor	25-160	25-160	63-133	47-142
Endosulfan sulfate	26-144	26-144	26-144	26-144
Endrin Ketone	10-200	10-200	10-200	10-200

Table 8
Minimum Retention Time (RT) Windows

Target Compound	RT Window Minimum
alpha-BHC	±0.05
beta-BHC	±0.05
gamma-BHC	±0.05
delta-BHC	±0.05
Heptachlor	±0.05
Aldrin	±0.05
alpha-Chlordane	±0.07
gamma-Chlordane	±0.07
Heptachlor epoxide	±0.07
Dieldrin	±0.07
Endrin	±0.07
Endrin aldehyde	±0.07
Endrin ketone	±0.07
DDD	±0.07
DDE	±0.07
DDT	±0.07
Endosulfan I	±0.07
Endosulfan II	±0.07
Endosulfan sulfate	±0.07
Methoxychlor	±0.07
Aroclors	±0.07
Toxaphene	±0.07
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

ATTACHMENT I

STANDARDS LOGBOOK

Standard Name: _____
Location: _____

Lot Code #: _____
Solvent Used: _____
Volume of Solvent Used: _____

Prepared By: _____
Date Prepared: _____
Expiration Date: _____

Stock Name	Vendor	Stock Lot # / Lot Code #	Logbook / Page #	Stock Conc	Amt Added	Final Vol
1.						
2.						
3.						
4.						
5.						

Calculations:

Analyte	Final Analyte Concentration	Analyte	Final Analyte Concentration

Comments: _____

Secondary Review By: _____ Date: _____

IEA, Inc. Form# QAL01100.NC

IEA Logbook# QA118A

ATTACHMENT II

Sample Request/Delivery Form

Date:_____ Department Name:_____ COCs Received by_____

#	Requestor	Project #	Sample #	Location	QC Level					Out	In	Matrix*
					I	II	III	CLP	COC			
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												

Matrix*: L = Leachate S = Soil W = Water O = Other

IEA, Inc. Doc #SMF00402.NC

IEA, Inc.
3000 Weston Parkway
Cary, NC 27513

Phone 919-677-0090
Fax 919-677-0427

IEA
An Aquarion Company

ATTACHMENT 6

Monroe,
Connecticut
203-261-4458

Schaumburg,
Illinois
847-705-0740

N. Billerica,
Massachusetts
508-667-1400

Whippany,
New Jersey
201-428-8181

SW-846 PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: INDUSTRIAL & ENVIRONMENTAL ANALYSTS

Lab Code: IEA Case No.: 2182-016 Method: 8080 SDG No.: 11245

Instrument ID: HP5890P6 GC Column:DB-1701

ID: 0.53 (mm) Date(s) Analyzed: 11/15/96

Level (x lvl1): lvl1:1.0 lvl2:2.0 lvl3:5.0 lvl4:10 lvl5:20

COMPOUND	RT OF STANDARDS LVL1	LVL2	LVL3	LVL4	LVL5	CURVE TYPE *
alpha-BHC	13.67	13.66	13.67	13.67	13.67	Averaged
beta-BHC	19.15	19.15	19.16	19.16	19.15	Averaged
delta-BHC	20.19	20.19	20.19	20.19	20.18	Averaged
gamma-BHC (Lindane)	15.55	15.55	15.55	15.55	15.55	Averaged
Heptachlor	16.46	16.45	16.46	16.46	16.46	Averaged
Aldrin	17.70	17.70	17.70	17.70	17.70	Averaged
Heptachlor epoxide	20.83	20.82	20.83	20.83	20.82	Averaged
Endosulfan I	21.95	21.95	21.95	21.95	21.95	Averaged
Dieldrin	23.43	23.43	23.43	23.43	23.43	Averaged
4,4'-DDE	22.89	22.89	22.89	22.89	22.89	Averaged
Endrin	24.17	24.17	24.17	24.17	24.17	Averaged
4,4'-DDD	25.81	25.80	25.81	25.81	25.83	Averaged
Endosulfan II	25.95	25.94	25.95	25.95	25.95	Averaged
4,4'-DDT	26.35	26.35	26.36	26.36	26.38	Averaged
Endo sulf/Methoxy	28.38	28.37	28.38	28.38	28.41	Averaged
Endrin ketone	29.66	29.66	29.66	29.66	29.68	Averaged
Endrin aldehyde	27.36	27.36	27.36	27.36	27.36	Averaged
Tetrachloro-m-xylene	9.29	9.29	9.29	9.29	9.29	Averaged
Decachlorobiphenyl	32.83	32.84	32.84	32.84	32.84	Averaged

* Origin included in linear, quadratic, and averaged curves

SW-846 PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: INDUSTRIAL & ENVIRONMENTAL ANALYSTS

Lab Code: IEA Case No.: 2182-016 Method: 8080 SDG No.: 11245

Instrument ID: HP5890P5 GC Column:RTX-35

ID: 0.53 (mm) Date(s) Analyzed: 11/15/96

Level (x lvl1): lvl1:1.0 lvl2:2.0 lvl3:5.0 lvl4:10 lvl5:20

COMPOUND	RT OF STANDARDS LVL1	LVL2	LVL3	LVL4	LVL5	CURVE TYPE *
alpha-BHC	14.32	14.32	14.32	14.31	14.31	Averaged
beta-BHC	16.63	16.62	16.62	16.62	16.62	Averaged
delta-BHC	18.29	18.29	18.29	18.29	18.28	Averaged
gamma-BHC (Lindane)	16.19	16.19	16.19	16.18	16.18	Averaged
Heptachlor	18.03	18.03	18.03	18.03	18.02	Averaged
Aldrin	19.56	19.56	19.56	19.55	19.55	Averaged
Heptachlor epoxide	22.07	22.06	22.06	22.06	22.06	Averaged
Endosulfan I	23.53	23.53	23.52	23.52	23.52	Averaged
Dieldrin	24.67	24.67	24.67	24.66	24.66	Averaged
4,4'-DDE	24.43	24.44	24.43	24.43	24.43	Averaged
Endrin	25.89	25.89	25.89	25.88	25.88	Averaged
Endosulfan II	26.55	26.55	26.55	26.54	26.54	Averaged
4,4'-DDD	26.40	26.40	26.40	26.40	26.40	Averaged
Endosulfan sulfate	28.12	28.12	28.12	28.12	28.12	Averaged
4,4'-DDT	27.45	27.45	27.45	27.45	27.44	Averaged
Methoxychlor	30.13	30.12	30.12	30.12	30.12	Averaged
Endrin ketone	30.51	30.51	30.51	30.50	30.50	Averaged
Endrin aldehyde	27.58	27.58	27.58	27.57	27.57	Averaged
Tetrachloro-m-xylene	11.06	11.06	11.06	11.05	11.05	Averaged
Decachlorobiphenyl	37.48	37.49	37.48	37.48	37.47	Averaged

* Origin included in linear, quadratic, and averaged curves

IEA Pesticide Standard Report

Sample Name : AR1660C/POC96-128 Report No : 2.00
esult File : /RESULT/P8091096_002.RES
olumn Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 1814 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 2 Bottle no. : 2

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline

#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.032	46811	VV	0.00000	
2	.63		.029	1975	VV	0.00000	
3	.87		.045	2980	PV	0.00000	
4	.96		.065	481	VB	0.00000	
5	1.14		.018	166	BB	0.00000	
6	1.83		.052	782	BV	0.00000	
7	2.27		.039	1222	PV	0.00000	
8	2.32		.049	1469	VV	0.00000	
9	2.90		.058	2610	VV	0.00000	
10	3.00		.058	1286	VV	0.00000	
1	3.17		.061	1421	PV	0.00000	
12	3.69		.078	9184	BV	0.00000	
13	4.61	4.62	.086	141818	BV	.20221	Tetrachloro-m-xylene
14	5.02		.044	496	VV	0.00000	
15	5.12		.070	4116	VV	0.00000	
16	5.23		.101	26621	VV	0.00000	
17	5.74		.130	31474	VV	0.00000	
18	6.21		.099	29963	PV	0.00000	
19	6.39		.130	186112	VV	0.00000	
20	7.00		.098	27956	VV	0.00000	
21	7.59		.116	410244	PV	0.00000	
22	7.95		.115	32380	VV	0.00000	
23	8.22		.117	112708	VV	0.00000	
24	8.35		.127	178739	VV	0.00000	
25	8.83		.145	96751	VV	0.00000	
26	9.11		.142	657197	VV	0.00000	
27	9.52		.192	373404	VV	0.00000	
28	9.86		.148	249127	VV	0.00000	
29	10.26		.207	635955	VV	0.00000	
30	11.04		.145	343425	VV	0.00000	
31	11.36		.192	314948	VV	0.00000	
32	11.93		.162	64320	VV	0.00000	
33	12.14		.206	43846	VV	0.00000	
34	12.51		.193	260697	VV	0.00000	
35	13.10		.171	32192	VV	0.00000	
6	13.38		.182	208548	VV	0.00000	

AR1016

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
37	13.63		.172	29840	VB	0.00000	
38	14.66		.182	6116	PV	0.00000	
39	15.29		.232	126190	PV	0.00000	
40	15.82		.238	339452	VV	0.00000	
41	16.22		.264	132842	VV	0.00000	
42	16.80		.255	557041	VV	0.00000	
43	17.60		.297	73758	VV	0.00000	AR1260
44	18.10		.286	90211	VV	0.00000	
45	18.64		.310	627731	VV	0.00000	
46	19.46		.431	360944	VV	0.00000	
47	20.08		.383	281006	VV	0.00000	
48	21.70		.429	742981	PV	0.00000	2386158 0.95
49	22.12 (1)		.310	428849	VV	0.00000	2511227
50	22.50		.328	256551	VV	0.00000	5.0%D
51	23.71		.380	71823	PV	0.00000	
52	24.58 (2)		.255	419084	VV	0.00000	
53	24.93		.255	231035	VV	0.00000	
54	25.22		.239	111202	VV	0.00000	
55	26.21		.235	81848	PV	0.00000	
56	26.71 (3)		.264	889567	VV	0.00000	
57	28.54		.128	102143	PV	0.00000	
58	28.81 (4)		.243	648658	VV	0.00000	
59	29.66		.129	5242	PV	0.00000	
60	30.76		.131	51642	PV	0.00000	
61	32.18		.138	131141	PV	0.00000	
62	33.45		.033	9606	PV	0.00000	
63	34.07	#34.09	.165	161184	PV	.21206	Decachlorobiphenyl

Total Area : 11501114 Total PPB : .414

Report Time : 1905 10Sep1996
Method : /METHOD/P8090396.MTH
esult File : /RESULT/P8091096_002.RES

IEA Pesticide Standard Report

Sample Name : AR1242C/POC96-133 Report No : 3.00
sult File : /RESULT/P8091096_003.RES
lumn Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 1911 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 3 Bottle no. : 3

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.031	44861	VV	0.00000	
2	.63		.030	2887	VV	0.00000	
3	.78		.028	308	PV	0.00000	
4	.87		.052	2476	VV	0.00000	
5	1.01		.109	2307	VV	0.00000	
6	1.14		.069	1238	PV	0.00000	
7	1.27		.114	487	VB	0.00000	
8	2.27		.047	1469	BV	0.00000	
9	2.31		.045	1362	VV	0.00000	
0	2.89		.068	2739	VV	0.00000	
11	2.99		.052	1025	VV	0.00000	
12	3.17		.067	1033	PV	0.00000	
13	3.69		.078	6856	VV	0.00000	
14	4.61	4.62	.087	150953	PV	.21523	Tetrachloro-m-xylene
15	5.12		.073	3948	VV	0.00000	
16	5.23		.101	23064	VV	0.00000	
17	5.74		.130	26276	VV	0.00000	
18	6.20		.098	24926	PV	0.00000	
19	6.39 1		.135	158594	VV	0.00000	
20	7.00		.101	23657	VB	0.00000	
21	7.59 2		.116	333530	BV	0.00000	
22	7.94		.114	25526	VV	0.00000	
23	8.21		.117	88620	VV	0.00000	
24	8.35		.129	146881	VV	0.00000	
25	8.83		.150	78766	VV	0.00000	
26	9.11 3		.149	522668	VV	0.00000	
27	9.52		.201	308339	VV	0.00000	
28	9.85		.154	203053	VV	0.00000	
29	10.25 4		.209	522237	VV	0.00000	
30	11.04		.149	297425	VV	0.00000	
31	11.35		.212	330180	VV	0.00000	
32	11.92		.156	82257	VV	0.00000	
33	12.14		.209	148034	VV	0.00000	
34	12.60		.300	480848	VV	0.00000	
5	13.08		.205	53893	VV	0.00000	

1537029 / 1413265 1.09

8.89% D

AR1242

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	13.64		.437	382891	VV	0.00000	
37	14.65		.297	70015	VV	0.00000	
38	15.20		.369	97373	VV	0.00000	
39	15.79		.343	111260	VV	0.00000	
40	16.78		.434	67533	VV	0.00000	
41	17.58		.485	83612	VV	0.00000	
42	18.64		.459	19932	VV	0.00000	
43	20.35		.424	35538	VV	0.00000	
44	21.72		.283	9418	PV	0.00000	
45	34.07		4.969	-4673524	PB	0.00000	

AR1242 (cont.)

Total Area : 4980297 Total PPB : .215

Report Time : 2001 10Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_003.RES

IEA Pesticide Standard Report

Sample Name : AR1248C/POC96-138 Report No : 4.00
esult File : /RESULT/P8091096_004.RES
olumn Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 2008 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 4 Bottle no. : 4

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

k#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.031	52814	VV	0.00000	
2	.63		.034	2840	VV	0.00000	
3	.78		.017	186	PV	0.00000	
4	.87		.041	2313	VB	0.00000	
5	1.14		.054	958	PV	0.00000	
6	1.20		.021	55	VV	0.00000	
7	2.27		.040	1158	BV	0.00000	
8	2.31		.049	1374	VV	0.00000	
9	2.90		.066	2545	PV	0.00000	
0	2.99		.047	1148	VV	0.00000	
11	3.17		.052	1076	VV	0.00000	
12	3.69		.079	5136	BV	0.00000	
13	4.61	4.62	.087	146153	PV	.20839	Tetrachloro-m-xylene
14	5.22		.122	2473	VV	0.00000	
15	5.63		.049	572	BV	0.00000	
16	6.24		.106	1948	BV	0.00000	
17	6.39		.114	13769	VV	0.00000	
18	7.00		.093	6101	BV	0.00000	
19	7.59 (1)		.114	166143	BV	0.00000	
20	7.94		.140	10484	VV	0.00000	
21	8.21		.103	37764	VV	0.00000	
22	8.35		.131	56318	VV	0.00000	
23	8.82		.175	30124	VV	0.00000	
24	9.10 (2)		.151	328063	VV	0.00000	
25	9.51		.235	206291	VV	0.00000	
26	9.85		.147	145601	VV	0.00000	
27	10.25 (3)		.197	766862	VV	0.00000	
28	11.03		.141	453878	VV	0.00000	
29	11.35		.179	435698	VV	0.00000	
30	11.92		.147	105294	VV	0.00000	
31	12.14		.195	220538	VV	0.00000	
32	12.59 (4)		.290	857006	VV	0.00000	
33	13.09		.197	83672	VV	0.00000	
34	13.64		.429	708447	VV	0.00000	
5	14.35		.223	45069	VV	0.00000	

2118074 / 1961573

1.08

8.0%D

AR1248

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	14.65		.258	136904	VV	0.00000	
37	15.19		.335	213777	VV	0.00000	
38	15.79		.291	253507	VV	0.00000	
39	16.77		.373	131162	VV	0.00000	
40	17.58		.432	182718	VV	0.00000	
41	18.63		.423	33395	VV	0.00000	
42	19.49		.292	14252	VV	0.00000	
43	20.34		.414	96597	PV	0.00000	
44	21.71		.335	27236	VV	0.00000	
45	22.48		.217	1727	PV	0.00000	
46	24.55		.285	-7954	PV	0.00000	
47	26.74		1.831	-22590	PV	0.00000	
48	34.07		1.154	-1114806	PB	0.00000	

AR1248 (Cont)

Total Area : 5991147 Total PPB : .208

Report Time : 2100 10Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_004.RES

IEA Pesticide Standard Report

Sample Name : AR1254C/POC96-143 Report No : 5.00
esult File : /RESULT/P8091096_005.RES
olumn Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 2105 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 5 Bottle no. : 5

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.038	31260	VV	0.00000	
2	.63		.030	1737	VV	0.00000	
3	.78		.038	718	PV	0.00000	
4	.87		.064	4169	VV	0.00000	
5	.96		.054	1518	VV	0.00000	
6	1.01		.090	2380	VV	0.00000	
7	1.14		.107	4456	VV	0.00000	
8	1.32		.140	4526	VV	0.00000	
9	1.39		.070	2064	VV	0.00000	
0	1.71		.315	7585	VV	0.00000	
11	1.83		.101	3539	VV	0.00000	
12	1.97		.120	2439	VV	0.00000	
13	2.27		.118	5271	VV	0.00000	
14	2.89		.060	1885	BV	0.00000	
15	3.17		.070	1031	PV	0.00000	
16	3.69		.077	4956	BB	0.00000	
17	4.61	4.62	.087	148200	BV	.21131	Tetrachloro-m-xylene
18	5.62		.097	1759	VV	0.00000	
19	6.39		.142	2826	BV	0.00000	
20	7.59		.103	7268	BB	0.00000	
21	8.22		.096	1746	BV	0.00000	
22	8.36		.130	2728	VB	0.00000	
23	8.92		.145	2988	BV	0.00000	
24	9.11		.171	15262	VV	0.00000	
25	9.42		.120	7497	VV	0.00000	
26	9.49		.183	10323	VV	0.00000	
27	9.85		.165	8304	VV	0.00000	
28	10.22		.159	338706	VV	0.00000	
29	11.03		.137	142392	VV	0.00000	
30	11.35		.197	82404	VV	0.00000	
31	11.92		.179	19915	VV	0.00000	
32	12.15		.189	60601	VV	0.00000	
33	12.51		.219	671475	VV	0.00000	
34	13.10		.175	120430	VV	0.00000	
35	13.38		.232	662577	VV	0.00000	

AR1254

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	13.65		.204	289417	VV	0.00000	
7	14.34		.206	44953	VV	0.00000	
8	14.65		.224	220692	VV	0.00000	
39	15.17		.294	457916	VV	0.00000	
40	15.79	2	.244	703992	VV	0.00000	
41	16.18		.247	108848	VV	0.00000	
42	16.78		.293	396633	VV	0.00000	
43	17.58	3	.335	468596	VV	0.00000	
44	18.07		.292	107120	VV	0.00000	
45	18.63		.329	335085	VV	0.00000	
46	19.49		.301	199356	VV	0.00000	
47	20.33		.504	304477	VV	0.00000	
48	21.12		.252	31788	VV	0.00000	
49	21.70	4	.414	645510	VV	0.00000	
50	22.45		.342	71113	VB	0.00000	
51	24.53		.351	204689	BV	0.00000	
52	25.22		.224	13029	VV	0.00000	
53	26.18		.246	59760	PV	0.00000	
54	26.70		.270	79869	VV	0.00000	
55	28.81		.119	26973	PV	0.00000	
56	34.07		.306	-306185	PB	0.00000	~~TCX~~ DCB

AR1254 (cont)

2480675 / 2408912 1.03

3.0%

Total Area : 7156751 Total PPB : .211

Report Time : 2204 10Sep1996
thod : /METHOD/P8090396.MTH
sult File : /RESULT/P8091096_005.RES

IEA Pesticide Standard Report

Sample Name : AR1221C/POC96-311 Report No : 7.00
sult File : /RESULT/P8091096_007.RES
lumn Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 2259 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 7 Bottle no. : 7

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

rk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.052	42420	BV	0.00000	
2	.79		.068	3551	PV	0.00000	
3	.89		.057	2057	VV	0.00000	
4	.95		.049	11572	VV	0.00000	
5	1.03		.109	21753	VV	0.00000	
6	1.32		.094	2098	PV	0.00000	
7	1.49		.200	3484	VV	0.00000	
8	1.55		.045	382	VV	0.00000	
9	1.69		.090	397	VB	0.00000	
0	1.92		.070	2155	BB	0.00000	
11	2.31		.065	2137	BB	0.00000	
12	2.76		.096	2698	BV	0.00000	
13	2.90		.094	1664	VV	0.00000	
14	2.99		.067	639	VB	0.00000	
15	3.67		.092	406317	BV	0.00000	
.6	4.26		.070	960	VV	0.00000	
17	4.61	4.62	.080	366844	BV	.51857	Tetrachloro-m-xylene
18	4.78		.135	172579	VV	0.00000	
19	5.12		.079	64584	VV	0.00000	
20	5.22		.112	163873	VV	0.00000	
21	5.73		.124	423621	VV	0.00000	
22	6.20		.103	254557	VV	0.00000	
23	6.39		.136	1076501	VV	0.00000	
24	7.00		.190	59386	VV	0.00000	
25	7.59		.124	285687	VV	0.00000	
26	7.82		.182	86378	VV	0.00000	
27	8.09		.122	105533	VV	0.00000	
28	8.21		.110	96192	VV	0.00000	
29	8.35		.151	152670	VV	0.00000	
30	8.83		.161	74568	VV	0.00000	
31	9.11		.157	349695	VV	0.00000	
32	9.52		.210	204362	VV	0.00000	
33	9.86		.168	129320	VV	0.00000	
34	10.26		.230	279513	VV	0.00000	
5	11.04		.156	138171	VV	0.00000	

AR1221

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	11.36		.238	156260	VV	0.00000	AR1221 (cont.)
37	11.93		.165	38943	VV	0.00000	
38	12.14		.222	66629	VV	0.00000	
39	12.60		.326	214669	VV	0.00000	
40	13.09		.190	24993	VV	0.00000	
41	13.65		.475	163679	VV	0.00000	
42	14.65		.295	32681	VV	0.00000	
43	15.20		.392	41352	VV	0.00000	
44	15.79		.336	42215	VV	0.00000	
45	16.80		.416	20801	VV	0.00000	
46	17.59		.406	20750	VV	0.00000	
47	20.32		.410	9772	PV	0.00000	
48	21.73		.179	2535	PV	0.00000	
49	34.07		1.239	-2697185	PB	0.00000	

Total Area : 5823594 Total PPB : .519

Report Time : 2349 10Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_007.RES

IEA Pesticide Standard Report

Sample Name : AR1232C/POC96-312 Report No : 8.00
esult File : /RESULT/P8091096_008.RES
olumn Type : DB-1701 30 Meter,0.53mm ID Inj. Vol. : 1 ul
Instrument : HP5890P8
Calculation : ExternalSTD
Run Time : 49.50 Mins. Injected on 2356 10Sep1996
Sequence File : /SEQUENCE/P8091096.SEQ
Subseq/Sample : 1/ 8 Bottle no. : 8

% Dil-Fact
100.00

Run Status : RunStatusOK
EndOffBaseline
NoReference

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
1	.54		.052	38121	BV	0.00000	
2	.79		.065	3408	PV	0.00000	
3	.89		.056	1683	VV	0.00000	
4	.95		.048	11600	VV	0.00000	
5	1.03		.108	21216	VV	0.00000	
6	1.34		.102	2266	PV	0.00000	
7	1.42		.086	1811	VV	0.00000	
8	1.50		.098	1542	VV	0.00000	
9	1.61		.060	327	VV	0.00000	
10	1.69		.077	237	VV	0.00000	
11	1.93		.069	1698	BB	0.00000	
12	2.31		.079	3321	BV	0.00000	
13	2.74		.145	3370	PV	0.00000	
14	2.90		.077	1062	VV	0.00000	
15	3.00		.062	368	VV	0.00000	
16	3.67		.095	130913	VV	0.00000	
17	4.11		.069	612	VB	0.00000	
18	4.61	4.62	.081	379783	PV	.53686	Tetrachloro-m-xylene
19	4.78		.148	71899	VV	0.00000	
20	5.12		.080	23231	VV	0.00000	
21	5.23		.114	77548	VV	0.00000	
22	5.74		.130	153014	VV	0.00000	
23	6.20		.104	104184	VV	0.00000	
24	6.39		.142	533284	VV	0.00000	
25	7.00		.159	60060	VV	0.00000	
26	7.59		.117	450735	VV	0.00000	
27	7.82		.090	31788	VV	0.00000	
28	7.94		.109	42245	VV	0.00000	
29	8.10		.093	45730	VV	0.00000	
30	8.21		.111	133416	VV	0.00000	AR1232
31	8.35		.142	232854	VV	0.00000	
32	8.83		.151	116881	VV	0.00000	
33	9.11		.151	744635	VV	0.00000	
34	9.52		.202	429069	VV	0.00000	
35	9.86		.158	284548	VV	0.00000	

IEA Pesticide Standard Report

Pk#	RT	ID-tm	Peak Width	Area	Code	PPB	Name
36	10.26		.213	654399	VV	0.00000	
37	11.04		.148	381490	VV	0.00000	
38	11.36		.214	409967	VV	0.00000	
39	11.93		.156	103707	VV	0.00000	
40	12.14		.208	169595	VV	0.00000	
41	12.61		.297	580569	VV	0.00000	
42	13.08		.200	60885	VV	0.00000	
43	13.65		.416	425152	VV	0.00000	
44	14.65		.293	68586	VV	0.00000	
45	15.20		.368	93417	VV	0.00000	
46	15.79		.339	109638	VV	0.00000	
47	16.78		.389	69985	VV	0.00000	
48	17.58		.503	67993	VV	0.00000	
49	18.64		.396	24307	VV	0.00000	
50	19.48		.456	12041	VV	0.00000	
51	20.34		.460	28246	VV	0.00000	
52	21.72		.347	12633	PV	0.00000	
53	22.09		.263	6355	VV	0.00000	
54	22.50		.258	2751	VV	0.00000	
55	24.58		.132	4523	PV	0.00000	
56	24.94		.258	4939	VV	0.00000	
57	25.25		.229	1284	VV	0.00000	
58	26.71		.122	7931	PV	0.00000	
59	28.82		.121	-6203	PV	0.00000	
60	34.07		.022	-49379	PB	0.00000	

AR1232 (cont.)

Total Area : 7438857 Total PPB : .537

Report Time : 0046 11Sep1996
Method : /METHOD/P8090396.MTH
Result File : /RESULT/P8091096_008.RES

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1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director _____

Quality Assurance Manager _____

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2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for removing sulfur from pesticide extracts.

3.0 SUMMARY OF METHOD

3.1 This method outlines the procedure for the removal of sulfur from a pesticide extract. Transfer the extract to a 40 ml vial and add TBA-Sulfite and 2-propanol. Then add water , shake, and remove hexane layer for analysis.

3.2 This SOP is based on the following method:

- USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) 2/88

4.0 INTERFERENCES

4.1 - Phthalate esters can interfere with pesticide determination; avoid any contact with plastic to best minimize this problem.

5.0 SAFETY

5.1 All compounds listed in Section 8 are toxic. Non-powdered polyvinyl gloves and safety glasses should be worn at all times when handling any of these solvents and compounds. Also, all work with these chemicals should be performed in a fume hood.

Material Safety Data Sheets (MSDS) for all chemicals used in operations are present in the laboratory for immediate access. Specific MSDS's should be reviewed for a complete knowledge of the toxicity and precautions of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

* Clear 40 ml VOA vials with Teflon-coated liner.

* Sample vials are never cleaned and reused.

7.0 APPARATUS AND MATERIALS

7.1

* 40 ml VOA vials with teflon-lined screw caps

* 0.8 ml and 1.8 ml autosampler crimptop vials and caps.

* Vial crimper

* Sterile Borosilicate glass transfer pipets/transfer bulbs.

* Safety glasses, non-powdered polyvinyl gloves, fume hood.

* Properly cooled refrigerators each for sample storage.

* Standard and Instrument Maintenance logbooks.

* Sample log-in books.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 **Solvents:** Hexane and 2-propanol should be pesticide grade or equivalent.

8.2 TBA-sulfite reagent: Using certified A.C.S. grade anhydrous Sodium Sulfite and Tetrabutylammonoium hydrogen sulfate, 97%, prepare as follows:

Dissolve 3.4 g of TBA into 100.0ml DI water and mix well. Rinse 3 times with 20 ml of hexane and discard hexane. Add 25 g of Sodium Sulfite, mix well, then add additional Sodium Sulfite until some remains undissolved.

8.3 Deionized water.

9.0 CALIBRATION

Not Applicable

10.0 QUALITY CONTROL

10.1 If only a partial set of extracts needs sulfur cleanup, set up a new reagent blank with 2 ml of hexane.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Transfer 2 ml of extract to a 40 ml VOA vial. Add 2 ml TBA-silfite reagent and 2 ml 2-propanol , cap the vial, and shake for at least 1 min. If the extract is colorless or if the initial color is unchanged, and if clear crystals are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 100 mg portions until a solid residue remains after shaking.

11.2 Add 5 ml DI water and shake for at least 1 min. Allow the extract to stand for 5-10 min. and use the hexane(top) layer for analysis.

12.0 CALCULATIONS

Not applicable

13.0 ACCEPTANCE OF DATA

13.1 Method or Reagent blank must contain no target compounds at a level above the quantitation limits of the analysis method.

14.0 REPORTING OF RESULTS

Not applicable

15.0 SUPPLEMENTAL DOCUMENTS

Not applicable

16.0 REFERENCES

16.1 Organic Analysis: Multi-Media, Multi-Concentration IFB-CLP, 2/88.

17.0 SUBSTANTIVE REVISIONS

Not applicable

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1.0 APPROVALS

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Laboratory Director

Quality Assurance Manager

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PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This digestion procedure is used for the preparation of surface water and ground water samples. The procedure is used to determine total recoverable metals or dissolved metals.

2.2 Samples prepared by this method may be analyzed by FLAA or ICP for the following:

Aluminum	Magnesium
Antimony	Manganese
Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Cadmium	Selenium
Calcium	Silver
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc
Lead	

2.3 The test codes for this document are MEPT51, METP52, and METP53.

3.0 SUMMARY OF METHOD

3.1 A mixture of HNO_3, HCl and material to be analyzed is refluxed in a 250 mL beaker. After the sample has been reduced to 15-20 mL the sample is brought to volume with DI water.

3.2 This method is based on EPA method 3005.

3.3 This method does not deviate from method 3005.

4.0 INTERFERENCES

4.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 10.0.

4.2 Contamination can be a problem in sample preparation. Trace grade acids are used to reduce this problem. These chemicals are described in section 8.0. In addition, a clean work area is very important.

5.0 SAFETY

5.1 This method uses concentrated acids. These chemicals can cause severe burns and eye damage. Always wear gloves when handling samples or reagents. Wear face shield, lab coat or apron to help protect against spills and splashes. Wear respirator to protect against acid fumes and fumes from samples. Keep hood closed while samples are digesting.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Samples should be received at pH <2.

6.2 Final digestates are to be placed in new 125 mL plastic bottles. Sample bottles are not to be reused.

6.3 Samples are stable when digestion is complete and need no preservation.

6.4 Holding time is 180 days.

7.0 APPARATUS AND MATERIALS

7.1 Equipment

7.1.1 Ventilation Hood

7.1.2 Hotplates

7.1.3 250 mL beakers

7.1.4 Watchglasses

7.1.5 Glass Rods

7.1.6 Graduated Cylinders

7.1.7 100 mL volumetrics

7.1.8 pH paper

7.1.9 Plastic bottles

7.1.10 Whatman number 42 filter paper

7.1.11 Funnels

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Barnstead deionized water, 16.6 megohm or higher

8.2 Concentrated Nitric Acid, Trace Grade

8.3 Concentrated Hydrochloric acid, Trace Grade

8.4 For preparation codes METP52 and METP53, no sample spikes or LCSW are performed as this is a dissolved undigested prep.

8.5 For preparation code METP51 the sample spike receives 1.00 mL of IEA-SPK-2 and 1.00 mL of IEA-SPK-1 for every 100 mL digested. The LCSW receives 1.00 mL of IEA-ICV-3 and 1.00 mL of IEA-ICV-1 for every 100 mL digested. Samples requiring B, Mo, Si, Sn, or Ti also receive 1.00 mL of IEA-ICV-2 for every 100 mL of LCSW digested.

9.0 CALIBRATION

9.1 No calibration is required for this method.

10.0 QUALITY CONTROL

10.1 Preparation blanks are performed to check for contamination. If a preparation blank is above the reporting limit and the samples are not a least ten times the preparation blank value then the samples in the batch are reprepped. The exception to this are if none of the samples are above the reporting limit then the batch is not reprepped.

10.2 A lab control sample is prepared to check that no analyte is lost or gained during the batch preparation. If the lab control sample does not meet the ± 20 % of the true value or the internally established control limits (whichever is more stringent), then the entire batch is reprepped. The exceptions to this are silver and antimony.

10.3 A duplicate sample is prepared and the RPD is calculated from the original sample. If the RPD is greater than 20 % and the sample is greater than five times the reporting limit the samples in that batch are flagged. If the sample is less than five times the reporting limit then the control limit is ± the reporting limit.

10.4 A sample spike is also performed each batch. This must agree within ± 25 % of the spiked value unless the original sample is greater than four times the spike level.

10.5 Control Charts are used to track the results of the lab control sample over time. Trends are noted to evaluate possible future problems such as analytes falling out of solution and the problem solved (i.e., replacing the solution or standard).

10.6 A sample spike duplicate is also performed each ICAP batch. This must agree within ± 20 % RPD with the original sample spike.

10.7 QC related Activities

Prep. Blank	Every Batch not to exceed 20 samples
Lab Control Sample	Every Batch not to exceed 20 samples
Sample Spike	Every Batch not to exceed 20 samples
Duplicate	Every 10 samples
Sample Spike Duplicate	Every Batch not to exceed 20 samples

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Using glass rods, take pH of sample and preserve if necessary.

11.2 Using a graduated cylinder, transfer a 100 mL representative aliquot of the well mixed sample to a 250 mL beaker and add 2 mL of the concentrated HNO_3 and 5 mL of concentrated HCl.

11.3 Place the beaker on a hotplate and cautiously evaporate to a 15-20 mL volume making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to dry.

11.4 Cool the beaker. Wash down the beaker walls and when necessary, filter sample to remove insoluble material. Before bringing the sample to volume add 3 mL of HNO_3 to the sample. Bring to volume with DI water to 100 mL in a volumetric flask. Place sample in a new 125 mL plastic bottle. The sample is now ready for analysis.

12.0 CALCULATIONS

12.1 None.

13.0 ACCEPTANCE OF DATA

13.1 Acceptance of the data is described in section 10.0

14.0 REPORTING OF RESULTS

14.1 Each log book page should have a date, analyst's name and batch number.

14.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, a blank sample and a laboratory control sample. All of these samples should be indicated in the log book.

14.3 Record pH, matrix, initial volume, and final volume for each sample.

14.4 Record lot number of all solutions and reagents added.

14.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 None

16.0 REFERENCES

16.1 Method 3005A, Acid Digestion of Waters for Total Recoverable Metals for Analysis by FLAA or ICP Spectroscopy, SW-846 Test Methods for Evaluating Solid Waste, July 1992 Revision.

16.2 Employee Chemical Safety Handbook, Industrial and Environmental Analysts, Inc., 1990.

17.0 SUBSTANTIVE REVISIONS

17.1 Test codes and sample spike and LCSW spiking levels were updated in this revision.

17.2 Addendum to Section 10.2: Control limit criteria added. Sections 10.6 and 10.7 were revised to include the MSD for ICAP analysis. Section 10.7 amended: Duplicate changed from every 20 to every 10 per NC. Section 11.4 was modified to include the addition of 3 mL HNO_3 for matrix match (sample and standard) purposes.

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1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

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PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This digestion procedure is used for the preparation of aqueous samples, EPTOX, Regulatory and Non-Regulatory TCLP extractions, and wastes that contain suspended solids for analysis by AA or ICP. The procedure is used to determine total metals.

2.2 Samples prepared by this method may be analyzed by FLAA or ICP for the following:

Aluminum	Lead
Arsenic	Magnesium
Barium	Manganese
Beryllium	Molybdenum
Cadmium	Nickel
Calcium	Potassium
Chromium	Selenium
Cobalt	Sodium
Copper	Thallium
Iron	Vanadium
	Zinc

2.3 The test codes for this document are METP15, METP50, and METP45.

3.0 SUMMARY OF METHOD

3.1 A mixture of HNO_3 and material to be analyzed is refluxed in a 250 mL beaker. This step is repeated with additional portions of HNO_3 until digestate is light in color or until its color has stabilized. After digestate has been brought to a low volume, it is refluxed with HCl and brought up to volume for the ICP. If the sample should go to dryness, it must be discarded and sample reprepared.

3.2 This method is based on EPA method 3010.

3.3 This method does not deviate from method 3010.

4.0 INTERFERENCES

4.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 10.0.

4.2 Contamination can be a problem in sample preparation and trace grade acids are used to reduce these problems. These chemicals are described in section 8.0. In addition, a clean work area is very important.

5.0 SAFETY

5.1 This method uses concentrated acids. These chemicals can cause severe burns and eye damage. Always wear gloves when handling samples or reagents. Wear face shield, lab coat or apron to help protect against spills and slashes. Wear respirator to protect against acid fumes and fumes from samples. Keep hood closed while samples are digesting.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Final digestates are to be placed in new 125 mL plastic bottles. Sample bottles are not to be reused.

6.2 Samples are stable when digestion is complete and need no preservation.

6.3 Holding time is 180 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 Equipment

7.1.1 Ventilation Hood

7.1.2 Hotplates

7.1.3 250 mL beakers

7.1.4 Watchglasses

7.1.5 Glass Rods

7.1.6 Graduated Cylinders

7.1.7 100 mL volumetrics

7.1.8 pH paper

7.1.9 Plastic bottles

7.1.10 Whatman number 42 filter paper

7.1.11 Funnels

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Barnstead deionized water, 16.6 megohm or higher

8.2 Concentrated Nitric Acid, Trace Grade

8.3 1:1 Hydrochloric acid, mix 8.1 with trace grade hydrochloric acid

8.4 For preparation codes METP52 and METP53 no sample spikes or LCSW are performed as this is a dissolved undigested prep.

8.5 For preparation code METP51 the sample spike receives 1.00 mL of IEA-SPK-2 and 1.00 mL of IEA-SPK-1 for every 100 mL digested. The LCSW receives 1.00 mL of IEA-ICV-3 and 1.00 mL of IEA-ICV-1 for every 100 mL digested. Samples requiring B, Mo, Si, Sn, or Ti also receive 1.00 mL of IEA-ICV-2 for every 100 mL of LCSW digested.

8.6 For preparation code METP15 samples spiklES are spiked in the leachate lab. LCSW samples receive 2.0 mL of TCLP SPIKE/LCSW STATE LEVELS for every 100 mL digested.

8.7 For preparation code METP45 sample spikes are spiked in the leachate lab. LCSW samples receive 1.0 mL of FEDERAL SPIKE A for every 100 mL of LCSW digested.

9.0 CALIBRATION

9.1 No calibration is required for this method.

10.0 QUALITY CONTROL

10.1 Preparation blanks are performed to check for contamination. If a preparation blank is above the reporting limit and the samples are not a least ten times the preparation blank value then the samples in the batch are reprepped. The exception to this are if none of the samples are above the reporting limit then the batch is not reprepped.

10.2 A lab control sample is prepared to check that no analyte is lost or gained during the batch preparation. If the lab control sample does not meet the $\pm$ 20 % of the true value then the entire batch is reprepped. The exceptions to this are silver and antimony.

10.3 A duplicate sample is prepared and the RPD is calculated from the original sample. If the RPD is greater than 20 % and the sample is greater than five times the reporting limit the samples in that batch are flagged. If the sample is less than five times the reporting limit then the control limit is $\pm$ the reporting limit.

10.4 A sample spike is also performed for each batch. This must agree within $\pm$ 25 % of the spiked value unless the original sample is greater than four times the spike level.

10.5 Control Charts are used to track the results of the lab control sample over time. Trends are noted to evaluate possible future problems such as analytes falling out of solutions and the problem solved, i.e., replacing the solution or standard.

10.6 A sample spike duplicate is also performed for each ICAP batch. This must agree within $\pm$ 20 % RPD with the sample spike.

10.7 QC related Activities

Prep. Blank	Every Batch not to exceed 20 samples
Lab Control Sample	Every Batch not to exceed 20 samples
Sample Spike	Every Batch not to exceed 20 samples
Duplicate	Every 10 samples
Sample Spike Duplicate	Every Batch not to exceed 20 samples

10.7.1 DEFINITIONS:

Batch: Every 20 samples leached, digested, or distilled per day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Using glass rods, take pH of sample and preserve if necessary.

11.2 Using a graduated cylinder, transfer a 100 mL representative aliquot of the well mixed sample to a 250 mL beaker and add 3 mL of the concentrated HNO_3.

11.3 Place the beaker on a hotplate and cautiously evaporate to a low volume (5 mL - 25 mL) making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to dry. Cool the beaker and add another 3 mL portion of concentrated HNO_3. Increase the temperature of the hotplate so that a gentle reflux action occurs.

11.4 Continue heating, adding additional acid if necessary, (no more than 10 mL) until the digestion is complete, (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Evaporate to a low volume not allowing any portion of the bottom of the beaker to go dry. Cool the beaker. Add 10 mL 1:1 HCl and warm the beaker for an additional 15 minutes to dissolve and precipitate or residue resulting from evaporation.

11.5 Wash down the beaker walls with DI water and when necessary, filter sample to remove insoluble material. Bring to 100 mL final volume with DI water. Place sample in a new 125 mL plastic bottle. The sample is now ready for analysis.

12.0 CALCULATIONS

12.1 None.

13.0 ACCEPTANCE OF DATA

13.1 Acceptance of the data is described in section 10.0

14.0 REPORTING OF RESULTS

14.1 Each log book page should have a date, analyst's name and batch number.

14.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, a blank sample and a laboratory control sample. All of these samples should be indicated in the log book.

14.3 Record pH, matrix, initial volume, and final volume for each sample.

14.4 Record lot number of all solutions and reagents added.

14.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 None

16.0 REFERENCES

16.1 Method 3010, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, September 1986.

16.2 Employee Chemical Safety Handbook, Industrial and Environmental Analysts, Inc., 1990.

17.0 SUBSTANTIVE REVISIONS

17.1 Test codes and sample spike and LCSW spiking levels were updated in this revision.

17.2 Addendum to Section 6.3 to clarify holding time. Section 8.1 ammended from 18.0 to 16.6 mega-ohm d.i. reagent water. Addendum to Section 10.6 to include definitions.

17.3 Revision to Section 2.3: Test codes METP17, METP47, METP51, METP52, METP53 omitted. Test code METP50 added. Sections 10.6 and 10.7 were updated to include the MSD for ICAP analysis. Section 10.7 revised: Duplicate changed from 20 samples to every 10 per NC.

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ATTACHMENT 10

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1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

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2.0 SCOPE AND APPLICATION

2.1 The purpose of this SOP is to describe the digestion procedure used to prepare aqueous samples, TCLP extracts and wastes that contain suspended solids for analysis by FLAA, ICAP, GFAAS or ICP/MS. Samples are digested using a microwave oven with temperature and pressure feedback.

2.2 This procedure is applicable to the following analytes:

Aluminum	Antimony	Arsenic	Barium
Beryllium	Cadmium	Calcium	Chromium
Cobalt	Copper	Iron	Lead
Magnesium	Manganese	Molybdenum	Nickel
Potassium	Selenium	Silver	Sodium
Thallium	Vanadium	Zinc	

2.3 The Test Codes applicable to this SOP are METP91, METP92 and METP93.

3.0 SUMMARY OF METHOD

3.1 A 45 mL aliquot of sample is digested using 5 mL of concentrated nitric acid in a Teflon microwave vessel. The sample is then cooled and placed in a plastic bottle.

3.2 This SOP is based on SW-846 Method 3015.

3.3 This SOP does not deviate from the above listed method.

4.0 INTERFERENCES

4.1 Samples with high organic matter will result in elevated vessel pressure. Venting will result in the loss of sample and/or analyte. The sample must not be vented. A smaller volume of sample diluted to 45 mL may be required to prevent this interference, resulting in elevated detection limits.

5.0 SAFETY

5.1 This procedure uses concentrated nitric acid. Care should be used when handling this acid and all chemicals. Strong acids cause severe burns to the skin and other soft tissues. Lab coats, gloves and safety glasses should be worn when using acids. Nitric acid fumes cause irritation of the eyes and nose and should be used in a fume hood.

5.2 Microwave ovens used in this method produce microwave radiation and should never be operated with the door open.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Samples should be collected in glass or plastic bottles.

6.2 Samples should be preserved with nitric acid.

6.3 Holding times for samples is 180 days from collection.

7.0 APPARATUS AND MATERIALS

7.1 1000 Watt Microwave Oven with computer control.

7.2 Teflon vessels with pressure relief caps, rupture disks and liners.

7.3 Sensor vessel and associated rupture disk.

7.4 Vessel Rack.

7.6 Portable hotplate.

7.7 Electronic Balance capable of weighing up to 900 grams to two decimal places.

7.8 50 mL graduated cylinder.

7.9 Plastic digestate storage containers.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Trace Metals Grade Concentrated Nitric Acid.

8.2 Ice.

8.3 DI water 16.6 megohm-cm or higher.

8.4 10% Aqua regia, Add 7.5 mL of HCl and 2.5 mL of HNO3 to a beaker. Dilute to 100 mL with DI water.

8.5 Matrix Spiking Solution, prepared as specified in the appropriate and corresponding referenced analytical SOP. (See Section 15.0.)

8.6 Laboratory Control Sample solution, prepared as specified in the appropriate and corresponding analytical SOP. (See Section 15.0.)

8.7 Reagent receipt is documented in the Chemical Receipt Logbook (Attachment I).

9.0 CALIBRATION

9.1 The temperature calibration is performed as follows, in accordance with manufacturer's instructions:

9.1.1 From the Main Menu select "Calibrate".

9.1.2 Select F2 for temperature calibration. Select the control vessel to calibrate (A for the first vessel, B for the second vessel, etc.) and press enter. The computer will prompt you to "Place the probe in 0 °C water and allow the temperature to stabilize for 10 seconds".

9.1.3 Place the probe in a Teflon beaker full of crushed ice and DI water. Ensure that the water is well mixed and full of ice. Wait for the reading to stabilize. Ten seconds after the reading stabilizes press enter. The zero point is now set.

9.1.4 The computer will now prompt you to "Place the probe in 100 °C water and allow the temperature to stabilize for 10 seconds". Place a hotplate near the microwave, but not inside the microwave. Bring a beaker of DI water to a vigorous boil. Place the probe in the beaker and wait for the temperature to stabilize. Ten seconds after the reading stabilizes press enter. The temperature calibration is now complete.

9.2 The pressure calibration is performed as follows, in accordance with the manufacturer's instructions:

9.2.1 From the Main Menu select "Run Method". Press F1 and select "CAL170". Make note of the data file used by the method. Fill the control vessel with 30 mL DI water and place rupture disk in place. Seal the vessel. Run the method by pressing F3. You will be prompted for the control vessel (A, B, etc.). When the vessel has been selected, the run will start. When the run is complete, return to the main menu.

9.2.2 From the Main Menu select "Calibrate".

9.2.3 Select F3 for pressure calibration. A message with detailed information regarding calibration will appear. Press enter. Select the control vessel and press enter. Select the data file from CAL170.MTH and press enter. If no errors are present the screen will display "Calibration Complete".

10.0 QUALITY CONTROL

10.1 Method Detection Limits (MDLs) are calculated in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET from samples prepared according to this SOP and analyzed by an acceptable corresponding method SOP.

10.2 Duplicates are prepared one per ten samples or per batch, whichever is more frequent.

10.3 Matrix Spikes are prepared one per twenty samples or per batch, whichever is more frequent. Spike 0.50 mL of spiking solutions into the matrix spike sample aliquot.

10.4 Matrix Spike Duplicates are prepared every twenty samples or per batch, whichever is more frequent. Spike 0.50 mL of spiking solutions into the matrix spike duplicate sample aliquot.

10.5 Preparation Blanks are prepared one per twenty or per batch, whichever is more frequent.

10.6 Laboratory Control samples are prepared one per twenty or per batch, whichever is more frequent. Spike 0.450 mL of spiking solutions into the LCSW.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Prepare the digestion vessel by heating 10% Aqua Regia in a beaker on a hotplate to 80° C. Rinse the Teflon liners with the hot Aqua Regia. Rinse the Teflon liners three times with DI water. **Never use a brush or wipe the liners with a paper towel as this may scratch the liner surface.** Liners that have scarring or cracks should be inspected by the supervisor and replaced if needed. Clean a 50 mL graduated cylinders in the same manner.

11.2 Weigh the complete digestion vessel, including the rupture disk to 0.01 g. Record the weight of the vessel and the vessel number in the Metals Digestion Logbook (Attachment II).

11.3 Shake the sample and measure exactly 45 mL of sample in a 50 mL graduated cylinder. Pour the sample into the digestion vessel and add 5 mL of concentrated nitric acid using the repipetor to each sample. Allow the samples to vent gases for five minutes or until the sample stops effervescing. Place the rupture disk in place and cap the vessel. Tighten the cap until resistance is felt and then tighten slighlty more (approximately an 1/8 of a turn). Select one representative sample for use in the control vessel. (Do not use the LCSW or the PBW.) Ensure that the outside of the vessels and the microwave cavity are dry.

11.4 Weigh the complete digestion vessel, including the rupture disk and sample to 0.01 g. Record the weight of the vessel and the vessel number in the Metals Digestion Logbook.

11.5 Place the vessels in the tray with the vent tube ends in the vent container. Plug the control vessel into the socket in the microwave cavity. The plug has a has a notch that, when introduced to the socket at the twelve o'clock position, will be in proper alignment with its counterpart. Close the door and ensure that the table rotates freely.

11.6 From the Main Menu select "Run Method". Press F1 to select the method. A list of methods will appear. Highlight and select "EPA3015". Press F2 to select the data file. Highlight "New" and type in the eight character batch ID (MMDDYYNN i.e., 01219501). The computer will prompt "Do you want to enter a notebook entry? (y/n)". Press "Y" and then enter. When the Notebook screen appears, enter the appropriate data. When complete press F10 to return to the Run Control screen. Ensure that the interlock button on the left side is out. You may stop the magnitron at any time by pressing the interlock button in. To start the run press F3. Select the control vessel in capital letters (A or B, see the control top). Press Enter and the run will begin. During the run press F4 to toggle between the graph and the run screen. Pressing F5 will toggle between the method screen and the run screen. F3 will stop the run. You will be prompted "Are you sure you want to STOP RUN (y/n):". Press y or n as needed. Press F10 to exit the screen when the run is complete.

11.7 Remove the vessels from the microwave by removing the entire rack after disconnecting the control vessel cable. Allow the samples to cool for five minutes in the rack. The samples may be cooled in a water bath, but ensure that the water is not above the threads. After the samples reach room temperature wipe the outside dry and weigh each vessel to 0.01 grams. Record the weight in the Metals Digestion Logbook. If more than 10% of the sample weight has been lost, the sample has vented and must be discarded.

11.8 Open each vessel one at a time and drain any condensation from the rupture disk to the liner. Remove the liner and transfer the sample to a new plastic bottle. Ensure that all condensation is transferred by turning the liner as it is poured. Do not rinse the liner to transfer the sample. Record the final volume in the Microwave Digestion Logbook as 50 mL.

11.9 Wash the vessels and liners with DI water. **DO NOT USE brushes to clean the liners.** Discard the rupture disks.

12.0 CALCULATIONS

12.1 Sample results are calculated as follows.

Preparation Factor = Final Volume/Initial Volume

Preparation Factor = 50 mL/45 mL

12.2 To calculate the loss of sample due to venting use the following formula.

$$\%\text{Loss} = \frac{(WB - WV) - (WA - WV)}{(WB - WV)} \times 100\%$$

WB = Weight of Sample and Vessel before digestion
WA = Weight of Sample and Vessel after digestion
WV = Weight of Vessel

13.0 ACCEPTANCE OF DATA

13.1 Sample data is acceptable for digestion if 10% or less of the sample is lost due to venting.

14.0 REPORTING OF RESULTS

14.1 Results are reported using ACS. This procedure is described in the SOP for ACS Data Reporting, IEA Doc# MES02900.NC.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.2 SOP for ACS Data Reporting, IEA Doc# MES02900.NC.

15.3 SOP for Metals Analysis by ICPMS Method 6020, IEA Doc# MES03200.NC.

15.4 SOP for SW-846 Method 6010 with TJA 61E ICAP Operation, IEA Doc# MES00801.NC.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, September 1994 Revision, USEPA, Method 3015.

16.2 QWave 3000 Operation Manual, Questron Corporation.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 10.2 revised to reflect duplicate frequency every 10 samples, or per batch. Revised 03/13/96.

Attachment I

Metals Lab Chemical Receipt Log

DATE	LOT #	VENDOR	REFERENCE #	GRADE	QTY	VOLUME/MASS	ANALYST

Secondary Review:_____ By:_____

IEA, Inc. Form# MEL01600.NC

Attachment II

Metal[illegible]gestion Log

IEA Sample #	EPA Sample #	pH	Microwave Digestion			Hotplate Digestion	Final Volume (mL)	Filter Data				Texture/Artifacts/Comments
			Vessel Wt (g)	Vessel + Sample Wt (g)	Wt After Digestion (g)	Sample Vol/Wt (mL/g)		Color		Clarity		
								Before	After	Before	After	

Batch #		Digestion Date		Matrix Spike #1 Lot #		LCS Lot #	
Method		HNO3 Lot #		MS #1 Spike Volume			
Dig. Analyst		HCL Lot #		Matrix Spike #2 Lot #			
Applicable SOP		H2O2 Lot #		MS #2 Spike Volume			
Matrix				Witness/Date			

Secondary Review By:_____ **Date:**_____

IEA, Inc. Form# MEL00703.NC

IEA Logbook# 10*

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director _____

Quality Assurance Manager _____

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2.0 SCOPE AND APPLICATION

2.1 The purpose of this SOP is to define the analysis of samples by cold-vapor atomic absorption for samples of a solid matrix. Samples are digested and then analyzed for mercury by reduction with stannous chloride.

2.2 The element determined is defined as "Total Metals" for a digested sample.

2.3 The IEA test code for this analysis is HG_54.

3.0 SUMMARY OF METHOD

3.1 This method is used to analyze samples that have been digested for mercury analysis. Samples are introduced into a flow cell with a nitrogen carrier gas where they absorb light at a characteristic wavelength. As the intensity of light absorbed is proportional to the concentration of mercury in the sample, these values are compared to a standard curve and the concentration of mercury determined.

3.2 This method is based on EPA method 7471.

3.3 This method deviates from the EPA method 7471 in that calibration curves are not constructed hourly. Instead, CCVs must pass or the run is terminated.

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide do not interfere with the recovery of mercury.

4.2 Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on mercury recovery.

4.3 High levels of free chlorine interfere with the analysis as chlorine absorbs at a wavelength of 253.7 nm also. Potassium permanganate oxidizes chlorides to chlorine. Therefore, additional potassium permanganate may be needed for seawaters, brines, etc. where salt content of the samples may be high. Hydroxylamine sulfate is added to the sample and the cell is purged with nitrogen before stannous chloride is added to the sample to remove any chlorine interferences.

4.4 Certain organic compounds interfere with mercury analysis. When organic interference is suspected, the sample should be analyzed without reagents to check for this interference.

5.0 SAFETY

5.1 Care should be used when working with concentrated acids and all protective gear worn. Safety glasses, lab coat and latex gloves are recommended to protect against glass breakage and sample spillage.

5.2 **Mercury is very toxic !** Care should be taken when handling mercury because elemental mercury is absorbed through the skin. Mercury is very volatile and is toxic if inhaled. All work should be vented to a hood.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 SAMPLE CONTAINERS: Sample containers may be either plastic or glass.

6.2 SAMPLE COLLECTION: Samples are collected in plastic or glass bottles.

6.3 SAMPLE PRESERVATION: None.

6.4 HOLDING TIMES: Samples are analyzed within 13 days after collection when collected in plastic containers and within 35 days after collection when collected in glass containers.

7.0 APPARATUS AND MATERIALS

7.1 Instrumentation - either of the following instruments may be used:

7.1.1 Perkin Elmer 5000 Spectrophotometer.

7.1.2 Mercury Monitor LDC 3200 with autosampler.

7.2 High purity nitrogen.

7.3 Calculator with linear regression function.

7.4 5 and 6 mL pipets and pipet bulb.

7.5 Peristaltic pump and tubing.

7.6 Tungsten-Lamp.

7.7 Analytical balance capable of weighing 0.20 grams, 0.01 g sensitivity.

7.8 Spatulas.

7.9 Plastic digestate storage bottle, with lid.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagent water - 16.6 megohm-cm, or higher, deionized.

8.2 Aqua regia, prepared immediately before use, three parts HCl and one part HNO_3

8.3 Sulfuric Acid, 0.5N, dilute 14.0 mL of concentrated sulfuric acid to 1 liter.

8.4 Stannous Chloride, 25 g to 250 mL in 0.5N H_2SO_4 this mixture should be stirred continuously as it is a suspension.

8.5 Sodium chloride-Hydroxylamine sulfate solution, dissolve 12 g of each in reagent water and dilute to 100 mL.

8.6 Potassium permanganate, 5% solution dissolve 5 g of potassium permanganate in 100 mL reagent water.

8.7 Potassium persulfate, 5% solution. Dissolve 5 g of potassium persulfate in reagent water and dilute to 100 mL.

8.8 Mercury Calibration Standard Preparation

8.8.1 Stock Mercury Standard is purchased at 1000 ppm from Ricca.

8.8.2 Dilute 1.0 mL of Stock Mercury Standard (Section 8.8.1) to 100 mL in a volumetric flask to prepare a 10 ppm standard solution.

8.8.3 Dilute 1.0 mL of the 10 ppm standard solution (Section 8.8.2) to 100 mL to prepare the 0.1 ppm intermediate standard.

8.8.4 Prepare a working curve from the 0.1 ppm intermediate standard as follows:

0.5 mL to the BOD bottle for the 0.5 ppb standard
1.0 mL to the BOD bottle for the 1.0 ppb standard
3.0 mL to the BOD bottle for the 3.0 ppb standard
5.0 mL to the BOD bottle for the 5.0 ppb standard
10.0 mL to the BOD bottle for the 10.0 ppb standard

8.8.5 For each of the standards prepared in Section 8.8.5, add enough reagent water to make the final volume 10 mL. Add 5 mL of aqua regia. Heat in a hot water bath for 2 minutes at 95°C. Cool the sample and add 50 mL of reagent water and 15 mL of $KMnO_4$. Heat the sample in the bath for 30 minutes. Add 6 mL of sodium chloride-hydroxylamine sulfate to each sample and allow the sample to aerate for thirty seconds.

8.8.6 Transfer the contents of the BOD bottle to a graduated cylinder and dilute to 150 mL with reagent water.

8.8.7 Transfer the contents of the graduated cylinder to a labelled digestate storage bottle.

8.9 Sample spikes receive 1.0 mL of the 10 ppb standard prepared in Sections 8.8.4 - 8.8.7 for every 100 mL of sample digested.

8.10 Initial Calibration Verification (ICV) Preparation

8.10.1 Stock Mercury Standard at 1000 ppm is purchased from Mallinckrodt.

8.10.2 The ICV is at 4 ppm and is prepared by adding 4.0 mL of Stock Mercury Standard (Section 8.10.1) to a BOD bottle and processing the aliquot as in Sections 8.8.5 - 8.8.7.

8.11 Mercury Certified Reference Material is purchased from Environmental Resource Associated to be used in preparation of the Laboratory Control Sample (LCSS).

8.12 All reagents and chemicals are documented in the Chemical Receipt Logbook (Attachment I). Chemical solutions for use in preparation and analysis for mercury are recorded in the Mercury Solutions Logbook (Attachment II).

9.0 CALIBRATION

9.1 Standards from Section 8.8 are to be analyzed in ascending order per the procedure described in Sections 11.2 and 11.3. After all standards are analyzed, the concentration and absorbance is entered into the linear regression function of the calculator. A correlation coefficient of $\geq$ 0.995 is an acceptable linear regression for the curve. Initial calibration acceptance criteria is $\pm$ 10% of the true value.

10.0 QUALITY CONTROL

10.1 Instrument detection limits are calculated based on the EPA SOW 3/90. A minimum of seven replicate standards are analyzed at three to five times the expected IDL on three nonconsecutive days and the average standard deviation multiplied by three to determine the IDL.

10.2 The Practical quantitation limit for this method is 0.10 ug/kg.

10.3 MDLs are determined in accordance with the SOP for Conducting MDL studies, IEA Doc# QAS02000.NET.

10.4 The Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB) and Preparation Blank consist of reagent water that is prepared as in Sections 8.8.5 - 8.8.7.

10.5 The LCSS is prepared and analyzed as in Section 11.0 using 0.20 - 0.25 g of Mercury Certified Reference Material (Section 8.11). The LCSS is not prepared in triplicate.

10.6 The Initial Calibration Verification (ICV) is prepared as in Section 8.10.

10.7 A Low Detection Limit Standard or Contract Required Standard (CRA) is prepared at 0.2 ppb by dilution and preparation of 0.2 mL of the 0.1 ppm standard (Section 8.8.3) as in Sections 8.8.5 - 8.8.7.

10.8 A sample duplicate is prepared as in Section 11.1 and is analyzed every 10 samples.

10.9 The 5 ppb standard prepared in Sections 8.8.4 - 8.8.7 is used as the Continuing Calibration Verification (CCV) and is analyzed every ten samples.

10.10 Sample spikes and sample spike duplicates are prepared as in Section 8.9.

10.11 The control limits, frequencies, and corrective actions for Quality Control Samples such as Sample Spikes, Duplicates, etc. are described in Table One.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Sample Preparation

11.1.1 Weigh a 0.2 g aliquot of sample in triplicate into clean 300 mL BOD bottles. Record the weight in the Mercury Digestion Logbook (Attachment III).

11.1.2 Add 5 mL of reagent water and 5 mL of aqua regia and heat in a hot water bath for 2 minutes at 95°C.

11.1.3 Cool the sample and add 50 mL of reagent water and 15 mL of $KMnO_4$. Heat the sample in the bath for 30 minutes.

11.1.4 Add 6 mL of sodium chloride-hydroxylamine sulfate to each sample and allow the sample to aerate for thirty seconds.

11.1.5 Transfer the contents of the BOD bottle to a graduated cylinder and dilute to 150 mL with reagent water.

11.1.6 Transfer the contents of the graduated cylinder to a labelled digestate storage bottle.

11.1.7 Sample spikes receive 1.0 mL of the 10 ppb standard prepared in Section 8.8.4 - 8.8.7 for every 100 mL of sample digested.

11.2 PE 5000 Analysis of Mercury

11.2.1 Place a cart in front of the PE 5000 and set up the stirrer and the peristaltic pump.

11.2.2 Attach a short length of Tygon tubing to the pump and attach the bubbler to the other end of the tubing. Run another length of tubing from the outlet on the bubbler to the inlet on the cell.

11.2.3 Make sure that all connections are air tight.

11.2.4 Place the bubbler in a digestate storage bottle containing about 150 mL of reagent water. Turn the pump on and set the air flow to deliver one cubic liter of air per minute.

11.2.5 Place a 1000 mL plastic bottle containing the stannous chloride on the stirrer with a stir bar in the bottle and turn the stirrer on. The stirrer should remain on at all times.

11.2.6 Turn the unit ON and turn the standby switch to RUN. Wait for the keyboard indicator to light up.

11.2.7 Plug in the mercury lamp and place it in the turret. Type the corresponding slot # for the lamp on the keyboard and press LAMP #. Type in the operating current on the mercury lamp and press LAMP MA.

11.2.8 To set the wavelength, type 253.7 and press PEAK. To set the slit width, type .7 and press SLIT HIGH.

11.2.9 To maximize the lamp, press SETUP and adjust the lamp until the reading on the instrument peaks. Then press SETUP again to exit. Press .5, t, CONT, REC ABS, ABS, AA.

11.2.10 Physically adjust the cell using the three silver knobs until the lowest reading is obtained. Press AZ to auto zero the instrument. Allow the instrument to stabilize for at least 10 minutes. The energy output should show at least 59.

11.2.11 Place the digestate storage bottle containing the sample to be run next to the bubbler on the cart. Pipet 5 mL of stannous chloride solution into the sample and **immediately** insert the bubbler into the sample. Do not disturb the sample during the read cycle.

11.2.12 Record the highest absorbance reading from the instrument readout in the Mercury Analysis Runlog (Attachment IV).

11.2.13 Leave the bubbler in the sample until all the mercury has been evolved and then place the bubbler back in the digestate storage bottle containing reagent water.

11.2.14 Repeat this process for each sample.

11.2.15 Calculate the concentration of mercury in each sample and record in the Mercury Analysis Runlog.

11.2.16 Upon completion of the run type 0 and press LAMP MA. Turn off the PE 5000 by switching the instrument to STANDBY and OFF. Clean up the area and remove pump tubing.

11.3 Mercury Monitor LDC 3200 Analysis of Mercury

NOTE: The operator of this instrument should familiarize himself/herself with WINDOWS software and with HGTALK software before operating this instrument.

11.3.1 Remove the Grey dryer tube (located on the front of the Mercury Monitor LDC 3200). Take off the bottom end of the tube and remove all the hardened magnesium perchlorate. Refill tube and reconnect to pneumatic tubes.

11.3.2 Turn on the computer. When "C:>" appears type in "Win" and hit "Return".

11.3.3 Power up the system components. The power switches for the Mercury Module and the Mercury Monitor LDC 3200 are located on the fronts of the components in the bottom right corner. The power switch for the Autosampler 2000 is located on the rear panel. Allow these to warm up for at least thirty minutes.

11.3.4 The flow meter is scaled from 0 to 10 and if nitrogen, argon or compressed air are used as the carrier gas, this should be set at three. This may be adjusted by rotating the "gas flow regulator". The reagent pressure gauge displays the reagent bottle pressure. This should be set from 4-6 psi. This is adjusted using the reagent regulator located on the rear panel of the unit.

11.3.5 A window directory will be on the screen. Place the arrow on "Cancel" and click once. Open a sequence file by placing the arrow on "Edit" at the top of the screen and clicking once. Place the arrow on "Edit Sequence" and click once.

11.3.6 A directory window containing sequences will be displayed. Choose AQUES.HSQ by clicking on it and then clicking once on "OK". A header will be displayed. Place the arrow on "Data File". Enter the new data file and click once on "OK".

11.3.7 The operator should enter his/her name and the date the samples were digested. Click once on "OK". Enter the samples in the correct vial positions. Move the arrow to "File",located in the top left corner of the screen and click once. Place the arrow on "Save" and click once. Open the "File" menu again and click on "Exit".

11.3.8 Move the arrow to "File" and click once. A window will appear that the file is about to be overwritten, press "OK". Another window will appear saying the results file will be overwritten. Click on "No". Enter a new results file name (use the same file name as the data file) and click once on "OK".

11.3.9 Load the standards and samples into their corresponding vial positions. Press F3 on the keyboard. Advance the autosampler to the correct position by pressing the "Advance Button", located on the rear of the autosampler, until the autosampler syringe is above the first standard. The system is now running.

11.3.10 The analysis must be documented in the Mercury Runlog (Attachment V). The LDC printout will be attached to a copy of the runlog and the analytical data filed by batch. The internal digestate COC is completed when digestates are removed for analysis and upon return to storage for all Level III analyses or samples designated for internal COCs.

12.0 CALCULATIONS

12.1 Sample Spike Recovery: $\% R = [(SSR - SR)/SA] \times 100$

SSR is Sample spike recovery in ug/L
SR is Sample result in ug/L
SA is Spike added in ug/L

12.2 $\% RPD = \left| (| A - B |)/ [(A + B)/2] \right| \times 100$

A is the sample result in ug/L
B is the duplicate result in ug/L

13.0 ACCEPTANCE CRITERIA

13.1	Initial Calibration	$\geq$ 0.995
	Initial Calibration Verification	$\pm$ 10%
	Continuing Calibration Verification	$\pm$ 20% of the true value
	Initial Calibration Blank	< PQL
	Continuing Calibration Blank	< PQL
	Method Blank	< Reporting Limit (0.10 ug/kg)
	Duplicates	$\pm$ 20% RPD
		$\pm$ Reporting Limit if sample concentration < 5 x Reporting Limit
	Laboratory Control Sample	95% Confidence Window

14.0 REPORTING OF RESULTS

14.1	Practical Quantitation Limit:	0.10 ug/kg
	Units of measure:	ug/kg
	Significant Figures:	5 figures
	Data Reporting:	Below quantitation limit: BQL
	QC Information included in report:	Level I QC: Blank values, sampling COCs Level II QC: Blank values, duplicate data (%RPD), spike data (% Recovery), sampling COCs Level III QC: Blank values, duplicate data (%RPD), spike data (%Recovery), ICV/CCV, LCSS, narrative, sampling and internal COCs

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 "Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, Method 7471.

17.0 SUBSTANTIVE REVISIONS

17.1 Test codes and spike levels were revised in this SOP. Also triplicate preps were added to match the method.

17.2 Section 8.1 amended from 18.0 to 16.6 mega-ohm d.i. reagent water. Addendum to 8.8.1 to include concentration of intermediate standard. Amendments to 8.8.1 to correct concentrations of standards. Addendum to Section 9.1 to include initial calibration acceptance criteria. Section 12.2 amended to more clearly express absolute value. Addendum to Section 13.0 to include initial acceptance criteria. Addendum to Table #1 to include initial calibration control limit.

17.3 Section 6.4 revised: Holding time changed from 28 days after collection to 13 days after collection for plastic bottles and 35 days after collection for glass. Addenda to Section 7.0: Section 7.1.1 added to include the Mercury Monitor 3200. Section 8.1 revised: megaohm changed to megohm-cm, or higher. Addendum to Section 8.0: Section 8.11 added to reference the Chemical Receipt Logbook and the Mercury Solutions Logbook. Addendum to Section 9.1: Correlation coefficient criteria defined. Initial calibration acceptance criteria clarified. Sections 10.2, 13.1, 14.1 and Table One revised to correct the PQL. Addenda to Section 10.0: Section 10.4 added to reference the SOP for Conducting MDL Studies. Addendum to Section 11.0: Section 11.2.12 added to reference the Mercury Runlog. Section 11.3 added to discuss mercury analysis by the Mercury Module 3200. Addendum to Section 13.1: Acceptance criteria for LCS, CCB and CCV defined. Addenda to Section 14.1: Level III QC added and sampling COCs added to Level I and Level II reporting. Addenda to Section 15.0: Section 15.1 added to reference the SOP for Conducting MDL Studies. Table #1 titled. Addendum to Table One: Sample Spike Duplicate control limit and corrective action listed. The frequency of QC samples added to Table #1. (05/10/96)

Quality Control Sample Control Limits and Corrective Action

Table #1

Quality Control Sample	Control Limit	Frequency	Corrective Action
IC	$\geq$ 0.995 correlation coefficient	Onset of Analysis	Recalibrate
ICV	$\pm$ 10%	Onset of Analysis	Recalibrate
ICB	$\pm$ 0.10 ug/kg	Onset of Analysis	Recalibrate
CRA	$\pm$ 20%	Onset of Analysis	None
CCV	$\pm$ 20%	1/10 samples	Rerun Samples
CCB	$\pm$ 0.10 ug/kg	1/10 samples	Rerun Samples
Duplicate	$\pm$ 20% RPD	1/10 samples	Flag Sample
Sample Spike	$\pm$ 25% Recovery	1/20 samples	Flag Sample
Sample Spike Duplicate	$\pm$ 20% RPD	1/20 samples	Flag Sample
Prep Blank	$\pm$ 0.10 ug/kg	1/20 samples	Reprep Samples
Lab Control Sample	95% Confidence Window	1/20 samples	Reprep Samples

Attachment I

Metals Lab Chemical Receipt Log

DATE	LOT #	VENDOR	REFERENCE #	GRADE	QTY	VOLUME/MASS	ANALYST

Secondary Review:_____ By:_____

IEA, Inc. Form# MEL01600.NC

Attachment II

Mercury Reagent Logbook
Metals Laboratory

Date	Analyst	Solution ID.	Description of Preparation	Expiration Date

Secondary Review By:_____ Date:_____

IEA, Inc. Form# MEF01100.NC

IEA Logbook# ME15*

Attachment III

Mercury Analysis Runlog
Instrument-*

Instrumental Analyst		SDG Number		Computer File Name	
Instrumental Analysis Date		SOP #		Correlation	

Sample ID#	Batch	Dilution Factor	Recovery	Comments

Secondary Review By:_____ Date:_____

IEA, Inc. Form# MEL00502.NC

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Cary, NC 27513

Phone 919-677-0090
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IEA
An Aquarion Company

ATTACHMENT 11

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1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

CONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This SOP defines the analysis of samples by inductively coupled argon plasma for samples of both solid, aqueous matrix, and TCLP extracts. Sample are introduced into a plasma where they emit light which is measured and the concentrations of those samples determined.

2.2 The elements determined are defined as "Total Metals" for a digested sample and as "Dissolved Metals" for a filtered and undigested sample.

2.3 The IEA test codes for this analysis are all test codes with the format EE_YX where EE is the element symbol from the periodic table and Y is 5 (5=SW846) and X is either 1, 2, 3, or 4 (1=Total Waters, 2=Dissolved lab filtered, 3=Dissolved field filtered, and 4=Soil samples). Also all EE_15 and EE_45 (15=NC TCLP and 45=Federal TCLP).

3.0 SUMMARY OF METHOD

3.1 Samples that have been previously digested for ICAP analysis are analyzed using a TJA Trace ICAP with an axial torch. Samples are nebulized with a meinhard nebulizer and a cyclonic spray chamber into an axial mounted plasma. Element-specific atomic-line emission spectra are dispersed by a grating polychrometer and the intensities of each line is measured by an array of photomultiplier tubes. Background correction is performed off peak center or by "Crawford/Kunselman" background correction technique.

3.2 This SOP is based on method SW-846 6010A.

3.3 This SOP deviates from method 6010A in respect to the following. RPD acceptance criteria for samples is five times the reporting limit instead of ten times the instrument detection limit.

4.0 INTERFERENCES

4.1 ICAP is subject to spectral interferences from overlap of the analytical line by other element emission lines, background continuum or recombination phenomena, overlap of molecular band spectra, and stray light from the line emission of high concentration elements. Background and stray light effects are corrected for by background correction as mentioned in section 3.1. Molecular band overlap is minimized by careful selection of emission lines and plasma operating conditions. Spectral overlap is corrected by Interelement Correction Factors (IECs), which are updated annually or as needed. Interelement Correction is performed automatically by the software before the raw data is generated.

4.2 Physical interferences occur due to problems with sample transport and nebulization due to viscosity and surface tension changes. These effects are overcome by use of a mass flow controller on the sample gas flow. Internal standards are also used to correct for physical interferences. Scandium and Yttrium are used as internal standards.

4.3 Chemical interferences are not normally present to much extent in ICAP analysis due to the high temperature of the argon plasma. Chemical interferences that may be present can be further minimized by careful selection of operating parameters and matrix matching.

5.0 SAFETY

5.1 Care should be used when working with concentrated acids and all protective gear worn. Safety glasses, lab coat and latex gloves are recommended to protect against glass breakage and sample spillage.

5.2 The torch produces intense ultraviolet light and should not be viewed without protection. Typical plasma temperature is between 6000 and 10,000 degrees Kelvin. Care should be used to insure that all parts are cool before attempting to service the instrument.

5.3 The instrument utilizes high voltages and service work should be restricted to qualified service personnel.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers: Sample containers may be either plastic or glass, 500 mL capacity or more and thoroughly rinsed and dried before sampling. Sample bottles are not to be reused.

6.2 Sample Collection: Samples are collected in plastic or glass bottles with nitric acid as a preservative to a pH of less than two.

6.3 Sample Preservation: Samples are preserved with 3 mL of 1:1 HNO_3 acid to a pH of less than two. Additional acid may be necessary for some samples.

6.4 Holding Times: 180 days from the date of collection.

7.0 APPARATUS AND MATERIALS

7.1 Thermo Jarrell Ash Trace 61E Vacuum ICAP.

7.2 High purity argon, welders grade or better

7.3 13 x 100 mm plastic test tubes

7.4 100 uL pipet

7.5 5.0 or 10.0 mL pipet

8.0 Reagents and Standard Preparation

8.1 Reagent water - 16.6 megohm or higher, deionized

8.2 1% HCl and 1% HNO_3 rinse, To a twenty liter carboy containing approximately five liters of 8.1 add 200 ml concentrated HCl and 200 mL concentrated HNO_3. Fill the remainder of the carboy with 8.1.

8.3 1000 mg/L Yttrium.

8.4 1000 mg/L Scandium.

8.5 CAL4 Stock, 1000 mg/L of Aluminum, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Manganese, Nickel, Selenium, Strontium, Thallium, Vanadium, and Zinc and 100 mg/L of Silver.

8.6 CAL3 Stock, 10000 mg/L of Calcium, Sodium, Potassium, and Magnesium.

8.7 CAL2 Stock, 1000 mg/L of Tin, Boron, Titanium, and Molybdenum.

8.8 CAL1 Stock, 1000 mg/L of Antimony.

8.9 10000 mg/L Calcium.

8.10 10000 mg/L Magnesium.

8.11 10000 mg/L Iron.

8.12 10000 mg/L Aluminum.

8.13 Concentrated HCl Acid.

8.14 Concentrated HNO_3 Acid.

8.15 Internal Standard Stock, To a 100.0 mL volumetric flask add 20 mL of 8.1 and 1.0 mL of 8.14. Pipet 50.0 mL of 8.3 and 10.0 mL of 8.4 into the flask. Dilute to volume with 8.1.

8.16 Working Standards are prepared as follows:

8.16.1 Blank, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.15. Dilute to the mark with 8.1.

8.16.2 CAL4, To a 200.0 mL volumetric flask containing 100 mL of 8.1 add 2.0 mL of 8.14. Next add 2.0 mL of 8.5 and swirl. Add 10.0 mL of 8.13 and swirl again. Dilute to the mark with 8.1.

8.16.3 CAL4D, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.15. Dilute to the mark with 8.1.

8.16.4 CAL2D, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.7. Dilute to the mark with 8.1. To a second 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14, 2.0 mL of 8.15 and 20.0 mL of the solution from the first flask. Dilute to the mark with 8.1. This second flask contains CAL2D.

8.16.5 CAL1, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.8. Dilute to the mark with 8.1.

8.16.6 CAL1D, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.16.5. Dilute to the mark with 8.1.

8.16.7 CAL3, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14, 2.0 mL of 8.15 and 2.0 mL of 8.6. Dilute to the mark with 8.1.

8.16.8 CAL5, To a 100.0 mL volumetric flask containing 50 mL of 8.1 add 5.0 mL of 8.13, 1.0 mL of 8.14, 1.0 mL of 8.15, 2.0 mL of 8.9, 2.0 mL of 8.10, 2.0 mL of 8.12 and 1.0 mL of 8.11. Dilute to the mark with 8.1.

8.2 All reagent receipt and preparation is documented in the Reagent Preparation and Receving Log (Attachment I).

8.3 All standards are documented in the Standards Preparation and Receiving Log (Attachment II).

9.0 CALIBRATION

9.1 Calibration standards are as follows, BLANK, CAL4D, CAL4, CAL2D, CAL1D, CAL1, CAL3, CAL5. Standards are analyzed in that order.

9.2 Working ranges and specific elements for each standard can be found in table two.

9.3 From the Analysis Menu press F3. The standards will be displayed on the screen. Place the cursor over the standard to be run and press F1 to run the standard after placing the probe in the standard. When the standard is complete press F9 to save the standard. Continue this process until all standards are run. After all standards are run press F9 to print the calibration. After the standards are printed press F9 to save the calibration. The standards are to be run in the order stated in 9.1. Internal standard must be added to CAL4 and CAL1 before these standards are run. All other standards have the internal already added.

9.4 1% Internal Standard (8.15) is added to all samples, QC, and standards.

9.5 Calibration correlation coefficient should be 0.995 or greater for all elements.

10.0 QUALITY CONTROL

10.1 MDLs are performed and calculated in accordance with DOC # QAS02000.NET.

10.2 Practical Quantitation Limits (PQL's) can be found in table two.

10.3 Daily Performance Checks are as follows.

10.3.1 Profile of the spectrum shifter using a five mg/L arsenic standard. Spectrum shifter position should be zero (0) before beginning the run.

10.3.2 Cadmium Background Ratio (BR) should be checked to insure proper instrument sensitivity. Cadmium BR should be 22.000. to 30.000.

10.4 Matrix Sample spikes (MS) are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent.

10.5 Matrix Sample Duplicates (MSD) spikes are to be performed one for every twenty

samples digested per matrix or one per batch, whichever is more frequent.

10.6 Continuing Calibration Verification (CCV) and Continuing Calibration Blanks (CCB) are to be performed every ten measurements and CCV and CCB are to be run at the end of the analytical run.

10.7 Duplicates are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent.

10.8 Serial dilutions are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent at a five fold dilution. Serial dilution results should be within ± 10% RPD.

10.9 Post spikes are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent. Post spikes should recover with 75 to 125 % of the known value.

10.10 Lab Contol Samples are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent.

10.11 QC activities are as follows:

ICV	Beginning of the run.
High Std Read-Back	Beginning of the run.
Interference Check	Beginning and end of run and every 8 hrs
Calibration Check Std. (CCV)	Every 10 samples and at the end of the run.
CCB	Every 10 samples and at the end of the run.
Method Blanks	One per Batch, per day, or every twenty samples
Matrix Spike	One per Batch, per day, or every twenty samples
Matrix Spike Duplicate	One per Batch, per day, or every twenty samples
Serial Dilution	One per Batch, per day, or every twenty samples
Post Spike	One per Batch, per day, or every twenty samples
Lab Control Sample	One per Batch, per day, or every twenty samples

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Autosampler tables are built by pressing the "Ctrl" key and the F3 key at the same time. The instrument will prompt for a macro name. Type AUTONAME and press return.

After the screen stops press Ctrl F3 again and this time type AUTOMAKE and press return. The instrument will build a autosampler table with all required QC, except the four required mixed standards. Return to the table using the appropriate keys and place the samples IDs in the sample slots. Insert the four mixed standards CAL4D, CAL1D, CAL2D, and CAL3 by pressing F1 and typing these names. Save these changes by pressing F9 until the table is saved.

11.2 To setup data storage perform the following steps. Press Ctrl F9, D, M, you will be prompted for a method name. Enter the method you are using. Press Enter followed by F3. Arrow down to analysis data file and enter the month, day, year, T, and the letter of the run (i.e. A for the first run of the day, B for the second, etc.). An example would be 82194TA for the first run on August 21, 1994. Press F9 until you exit the method. Press Ctrl F9, C, I followed by F9 until you reach the remote data screen. Arrow down to the remote data file and enter the same file name here. Press F9 until the main screen returns. Leave the software by pressing X, Q. Type CD\STATION\DATA\ANALYTIC and return. Rename the SAMPLES.DBF file to XXXXXXX.SAM using the command REN SAMPLES.DBF XXXXXXXX.SAM, where XXXXXXXXX is some user chosen name. Return to the software by typing WIN followed by return.

11.3 The analysis menu may be reached by pressing Ctrl F9, O, A and entering the method to be run followed by the return key.

11.4 To profile the instrument press the F5 key followed by the F3 key while aspirating a five mg/L solution of arsenic. Next press F1 to start the profile. If the profile is acceptable (between - 0.10 and 0.10) press F9 twice to save the profile. If the profile is not acceptable press F1 to calculate the required SS position and turn the dial on the instrument to this position. Repeat the profile procedure.

11.5 Calibrate as described in section 9.3.

11.6 To load the autosampler tray pour the solution into the QC cups (the L shaped rack). These solutions already have the internal standards present. To load the samples pipet 100 uL of internal standard into the 15 mL test tube and 10 mL of sample. Mix. Place in the correct rack position.

11.7 To run samples press F9 from the analysis menu followed by F1 to start the autosampler tray. Analysis will proceed automatically.

11.8 All analyses are documented in the instrument runlog for the ICP61T (Attachment III).

12.0 CALCULATIONS

12.1 $RPD_{duplicate} = \left| \frac{S - D}{(S+D)/2} \right| \times 100$

12.2 $RPD_{MS/MSD} = \left| \frac{MS - MSD}{(MS+MSD)/2} \right| \times 100$

12.3 Recovery = [(PS - S)/SA] x 100

12.4 $RPD_{SERIALDILUTION} = \left| \frac{S - SD}{S} \right| \times 100$

where:

S = Sample result
SD = Serial Dilution result
MS = Matrix Spike result
MSD = Matrix Spike Duplicate result
SA = Spike Added
PS = Post Spike result
D = Duplicate Result

13.0 ACCEPTANCE OF DATA

13.1 Duplicate control limit is as follows:

13.1.1 ±20% RPD for samples greater than or equal to five times the reporting limit.

13.1.2 ±the reporting limit for samples less than five times the reporting limit.

13.2 Post Spike control limit is 75 to 125% of known value.

13.3 Serial Dilution control limit is ± 10% RPD.

13.4 Control limit for MS and MSD is ± 20% RPD.

13.5 Matrix Spike control limit is 80 to 120 % recovery.

13.6 Method Blanks must be within ± PQL.

13.7 ICV, CCV must be within ± 10% of known value.

13.8 High standard read back must be within ± 5% of known values.

14.0 REPORTING OF RESULTS

14.1 Results are reported as follows:

Level I:	Sample Results, Method Blank Results
Level II:	Level I plus sample spike recovery and duplicate RPD summaries
Level III:	Level II plus ICV and CCV recovery and internal COCs

14.2 Results are reported to three significant figures.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, Septmeber 1994 Revision, USEPA, Method 6010A.

17.0 SUBSTANTIVE REVISIONS

17.1 Original Issue (11/30/94).

17.2 Sections 8.2 and 8.3 and 11.8 added. Table two revised to include soil units. Table three added. Section 16.1 corrected. Section 17.1 dated. (12/10/96)

TABLES

Table One

Std. Name	Elements Used from This Standard	Concentration (ug/L)
BLANK	All Elements	0.0
CAL4D	Al, As, Ba, Be, Cd, Cr, Co, Cu, Fe, Mn, Ni, Ag, Tl, V, Zn, Li, Sr, Pb, Se	1000, except Ag (100)
CAL4	Al, As, Ba, Cr, Co, Cu, Fe, Mn, Ni, Ag, Tl, V, Pb, Se	10000, except Ag (1000)
CAL2D	Mo, Sn, Ti, B	1000
CAL1D	Sb	1000
CAL1	Sb	10000
CAL3	Na, K, Ca, Mg	100000
CAL5	Ca, Mg, Al, Fe	100000 Fe,200000 Al,Ca,Mg

Table Two*

Element(s)	PQLs	
	Waters (ug/L)	Soils (mg/Kg)
Ca, Mg, K, Na	5000	500
Se, Cd, Be	5.0	0.5
As, Tl, Ag, Cr	10.0	1.0
Cu	25.0	2.5
Co, Mo, V, Ti, Sn, B, Sr, Li	50.0	5.0
Zn	20.0	2.0
Ni	40.0	4.0
Sb	6.0	0.6
Pb	3.0	0.3
Al, Ba	200.0	20
Fe	100.0	10
Mn	15.0	1.5

*PQLs provided are the standard IEA deliverable and may be adjusted based on client specified requests.

Table Three
Recommended Wavelenghths

Element	Wavelength (nm)
Aluminum	308.205
Antimony	206.838
Arsenic	189.042
Barium	493.409
Beryllium	313.042
Boron	249.678
Cadmium	226.502
Calcium	317.933
Chromium	267.716
Cobalt	228.616
Copper	324.754
Iron	271.441
Lead	220.353
Lithium	670.784
Magnesium	279.078
Manganese Manganese (2nd Line)	257.610 344.199
Molybdenum	202.030
Nickel	231.604
Potassium	766.491
Selenium	196.026
Silver	328.068
Sodium	330.232
Strontium	421.552
Thallium	190.864
Tin	189.989
Titanium	334.941
Vanadium	292.402
Zinc	213.856

ATTACHMENT I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
			R			
			R			
			R			
			R			
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			R			
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Secondary Review By:_____ Date:_____

ATTACHMENT II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

condary Review By:_____ **Date:**_____

ATTACHMENT III

ICP Runlog
Instrument-61T

Computer File Name		**Preparation Date**	
Analyst		**Instrumental Analysis Date**	
SDG Number		**Method Number**	

Sample ID#	Batch #	Initial Weight or Volume	Final Volume	Dilution Factor	Comments

Secondary Review By:_____ Date:_____

IEA, Inc. Form# MEF00300.NC

IEA Logbook# ME*

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IEA
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ATTACHMENT 12

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1.0 APPROVALS

The signature of the following individuals indicates that this Standard Operation Procedure (SOP) is complete and meets the requirements specified in Corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This method describes the procedure for the analysis of aqueous and semi-aqueous samples for acidity, expressed as $CaCO_3$. The applicable range is 10 to 1000 mg/L.

2.2 This method is applicable to aqueous samples, including surface and groundwaters, and domestic and industrial wastes.

2.3 This method is applicable to Test Code ACD_01.

3.0 SUMMARY OF METHOD

3.1 The pH of the sample is determined and a measured amount of standard acid is added, as needed, to lower the pH to 4 or less. Hydrogen peroxide is added and the solution is boiled for several minutes, cooled, and then titrated electrometrically with standard alkali to pH 8.2.

3.2 This method is based on Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 402, and on EPA Method 305.1.

3.3 This method does not deviate from the above methods.

4.0 INTERFERENCES

4.1 Suspended solids or precipitates formed during titration may cause a sluggish electrode response. Titrant may be added slowly dropwise as the endpoint pH is approached, to offset this interference.

5.0 SAFETY

5.1 Hydrogen peroxide is a strong oxidizer. Gloves should be worn when handling, and great care must be taken when using it in the vicinity of a hot plate.

5.2 Goggles or face shield, lab coat, and gloves are necessary if preparing titrants from concentrated sulfuric acid and sodium hydroxide pellets.

5.3 Potassium hydrogen phthalate may cause skin and respiratory irritation. Gloves are recommended when handling; avoid breathing dust.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be glass or plastic, thoroughly rinsed and dried before use, and are not to be reused.

6.2 Sample collection: Samples are collected without preservatives. Samples are packaged and sent to the laboratory and stored at 4 °C until analysis.

6.3 Sample preservation is not necessary for this analysis.

6.4 The holding time for acidity analysis is 14 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 pH meter with combination electrode and temperature probe

7.2 25 mL buret, graduated in 0.1 mL increments

7.3 125 mL Erlenmeyer flasks

7.4 250 mL beakers

7.5 50 mL graduated cylinders

7.6 Hot plate

7.7 Assorted beakers, volumetric pipets, volumetric flasks, watch glasses

7.8 Stir plate, stir bars, magnetic stir bar retriever

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent water, 16.6 megohm or higher, deionized

8.1.2 Potassium hydrogen phthalate (KHP), ACS grade, dried

8.1.3 Phenolphthalein indicator

8.1.4 Ethanol 95%, not denatured

8.1.5 Sodium hydroxide pellets (NaOH), ACS grade

8.1.6 Optional: Purchased solutions of NaOH, 0.1 N and 0.02 N

8.1.7 Concentrated sulfuric acid (H_2SO_4), ACS grade

8.1.8 Optional: Purchased solutions of H_2SO_4, 0.1 N and 0.02 N

8.1.9 pH 4, pH 7, and pH 10 buffers, purchased

8.1.10 Hydrogen peroxide (H_2O_2), 30% solution

8.2 Reagent prep:

8.2.1 Phenolphthalein indicator solution: Dissolve 0.5 g phenolphthalein in 50 mL 95% ethanol and 50 mL DI water.

8.2.2 Standard NaOH titrant, 0.1 N: Dissolve 4 g of NaOH pellets in DI water and bring to 1 liter volume.

8.2.3 Standard NaOH titrant, 0.02 N: Dilute 400 mL of 0.1 N NaOH solution to 1 liter.

8.2.4 Standard H_2SO_4 titrant, 0.1 N: Dilute 2.8 mL of concentrated H_2SO_4 to 1 liter.

8.2.5 Standard H_2SO_4 titrant, 0.02 N: Dilute 400 mL of 0.1 N H_2SO_4 solution to 1 liter.

8.3 All reagent receipt and preparation is recorded in the Reagent Receipt and Preparation Logbook (Attachment I).

8.4 Titrant standardization:

8.4.1 0.1 N standard NaOH:

8.4.1.1 Weigh 0.205 g dried KHP into a 125 mL Erlenmeyer flask; record weight to 0.1 mg. Add 50 mL DI water; swirl to dissolve.

8.4.1.2 Prepare two more KHP solutions as above.

8.4.1.2.1 Add 50 mL DI water to a 125 mL Erlenmeyer flask as a Blank. Add 1 to 2 drops of phenolphthalein and a stir bar to each flask.

8.4.1.2.2 Rinse and fill a 25 mL buret with 0.1 N NaOH solution. Record the initial volume.

8.4.1.2.3 Titrate the Blank and each KHP solution until the pink endpoint persists for 30 seconds. Record the final volume.

8.4.1.2.4 Calculate the normality of the NaOH solution as in Section 12.1.

8.4.2 0.02 N NaOH: Standardize as for 0.1 N NaOH, using 0.041 g dried KHP.

8.4.3 0.1 N H_2SO_4:

8.4.3.1 Pipet 10.0 mL of standardized 0.1 N NaOH solution into each of three 125 mL Erlenmeyer flasks. Add 40 mL of DI water, 1 to 2 drops of phenolphthalein, and a stir bar to each flask.

8.4.3.2 Rinse and fill a 25 mL buret with 0.1 N H_2SO_4 solution. Record the initial volume. Titrate each solution to a clear endpoint. Record the final volume.

8.4.3.3 Calculate the normality of the H_2SO_4 solution as in Section 12.2.

8.4.4 0.02 N H_2SO_4: Standardize as for 0.1 N H_2SO_4, using 10.0 mL of 0.02 N NaOH solution.

8.5 Record all solution standardizations in the Titrant Standardization Logbook (Attachment II).

8.6 Standard solutions:

8.6.1 Initial and Continuing Calibration Standards: Purchased from Hach Co., Cat # 14330-10, 25,000 mg/L as CaCO3.

8.6.1.1 Dilute the 10 mL voluette to 500 mL, to make a 500 mg/L calibration standard.

8.6.1.2 Dilute 50 mL of this solution to 250 mL, to make a 100 mg/L calibration standard.

8.6.2 Matrix spiking standard: 500 mg/L calibration standard.

8.6.2.1 Add 3.0 mL to 50 mL of a sample with an initial pH below 4.0, to constitute a 30 mg/L spike.

8.7. All Standards are documented in the Standards Receipt and Preparation Logbook (Attachment III).

9.0 CALIBRATION

9.1 pH meter calibration: Calibrate using pH 4 and pH 10 buffers. The efficiency (slope) must be greater than 0.90; preferably greater than 0.95.

9.1.1 Record the pH calibration in the logbook for potentiometric analysis of pH (Attachment IV).

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated based on the 40 CFR, Part 136, Appendix B definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 The practical quantitation limit for this method is the same as the method detection limit.

10.3 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), Section 8.6.1.

10.4 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Matrix Spike	Every 20 samples, per batch
Duplicate	Every 20 samples, per batch

10.4.1 DEFINITIONS:

Batch: Every 20 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Analytical Procedure

11.1.1 Standardize the pH meter, using the pH 4 and pH 10 buffers; record the Efficiency. Read and record the pH 7, pH 10, and pH 4 buffers.

11.1.2 Add 50 mL of sample to a 250 mL beaker; add stir bar. Place the beaker on a stir plate and adjust stirring. Place pH probe in sample. Allow the pH to stabilize; record the initial pH.

11.1.3 If the sample pH is above 4.0, add standardized 0.02 N H_2SO_4 in 5.0 mL increments to lower the pH to 4.0 or less.

11.1.4 Add 5 drops of hydrogen peroxide and cover with a watch glass. Place the beaker on a hot plate.

11.1.5 Heat the sample to boiling and continue boiling for 2 to 4 minutes. Remove the beaker from the hot plate and allow the sample to cool to room temperature.

11.1.6 Rinse and fill a 25 mL buret with standardized 0.02 N NaOH. Place the beaker on a stir plate and adjust stirring. Allow the pH to stabilize.

11.1.7 Record the initial volume of titrant in the Titrimetric Analysis Logbook (Attachment V). Add titrant dropwise to raise the pH to 8.2. Record the final volume of titrant in the Titrimetric Analysis Logbook.

11.1.8 For ICV and CCV, pour 50 mL of calibration verification standard into a 250 mL beaker. Analyze as for sample.

12.0 CALCULATIONS

12.1 $$N\ NaOH = \frac{(g\ KHP)(1000)}{(204.2)(V_t - V_b)}$$

where V_t = final volume of titrant - initial volume of titrant
V_b = volume of titrant used for the Blank

12.2 $$N\ H_2SO_4 = \frac{(N\ NaOH)(mL\ NaOH)}{V_t}$$

Where V_t = final volume of titrant - initial volume of titrant

12.3 Analyte:

$$\frac{[(V_t)(N_t) - (V\ H_2SO_4)(N\ H_2SO_4)](meq\ wt)(1000)}{mL\ sample} = mg/L\ CaCO_3$$

where: V_t = volume of titrant used for sample
N_t = normality of titrant
$V\ H_2SO_4$ = volume of H_2SO_4 added to reduce pH to 4 or less
$N\ H_2SO_4$ = normality of H_2SO_4

meq wt = 50.04 mg/meq

12.4 ICV and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{100 mg/L}} \times 100 = \%\ \text{Recovery}$$

12.5 Relative Percent Difference:

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

12.6 Matrix Spike %Recovery

Spiked Sample Result - Sample Results ÷ Spike Amount X 100%

13.0 ACCEPTANCE CRITERIA ACIDITY ANALYSIS:

ICB, CCB	< 10 mg/L (MDL)
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % or ± 10 mg/L (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level 1 QC: Blank values.
Level 2 QC: Blank values, Duplicate data (%RPD).
Level 3 QC: Blank values, Duplicate data (%RPD), matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS

16.0 REFERENCES

16.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, p 265, Method 402(4d), 1985.

16.2 Methods of Chemical Analysis of Water and Wastes, Second Edition, Method 305.1, June 1982.

17.0 SUBSTANTIVE REVISIONS

17.1 Original issue (12/09/96).

ATTACHMENT I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
			R			
			R			
			R			
			R			
			R			
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			R			
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			R			
			R			
			R			
			R			

Secondary Review By:_____ Date:_____

IEA, Inc. Form# CVL01200.NC

IEA Logbook# CV17

ATTACHMENT II

TITRANT STANDARDIZATION

DATE:_____ Analyst:_____

TITRANT:_____ Date Prepared:_____

STANDARD:_____ Std Concentration:_____ Std #:_____

ID	mL Std	Titrant V_I (mL)	Titrant V_F (mL)	Titrant V_T (mL)	Conc (meq/mL)
Blk					
A					
B					
C					
Ave. A, B & C					

Concentration:

meq/mL =

DATE:_____ Analyst:_____

TITRANT:_____ Date Prepared:_____

STANDARD:_____ Std Concentration:_____ Std #:_____

ID	mL Std	Titrant V_I (mL)	Titrant V_F (mL)	Titrant V_T (mL)	Conc (meq/mL)
Blk					
A					
B					
C					
Ave. A, B & C					

Concentration:

meq/mL =

Comments:_____

_____

Secondary Review:_____ Date:_____

ATTACHMENT III

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

econdary Review By:_____ Date:_____

ATTACHMENT IV

POTENTIOMETRIC ANALYSIS

Method Name:_____ Date:_____ Analyst:_____

DAILY STANDARDS

Standard ID	Stock Lot#	Stock Vol.	Stock Conc.	Diluent	Final Vol.	Final Conc.
ICV						
LCS/QC Check						
Working Standard						

STANDARD CURVE

Samples/Standards Final Volume Required:_____mL

Standard ID	Std Concentration	pH Reading

Curve Correlation Coefficient (must be $\geq$ 0.995):_____ Slope:_____

Sample ID	Volume Used (mLs)	pH Reading	Temperature °C	Final pH Units	QC Sample (RPD)
ICV					
LCS/QC CHECK					
CCV					

CALCULATIONS:

$$RPD = \frac{|(\text{Sample} - \text{Sample Dup.})|}{\text{Avg., Sample} + \text{Dup.}} \times 100$$

Comments:_____

_____

Secondary Review By:_____ Date:_____

ATTACHMENT V

TITRIMETRIC ANALYSIS

Method Name:_____ DATE:_____ Analyst:_____

Daily Standards and Spike Preparation							
Standard ID	Stock Lot#	Stock Conc	Stock Vol	Diluent	Final Volume	Final Conc	Std #
ICV/QC Check							
Working Standard							
Spike							

TITRANT:_____ Normality:_____ Reference:_____

Sample ID	mLs Sample	Titrant V_I	Titrant V_F	Titrant V_T	ug/mL	%Rec. / RPD
ICV						
ICB						

CALCULATIONS:

$$RPD = \left| \frac{(Sample - Sample\ Dup.)}{Avg., Sample + Dup.} \right| X\ 100 \quad \% \text{ Recovery} = \frac{\text{Spiked Sample Result - Sample Result}}{\text{Spike Concentration}} X\ 100$$

Concentration:

ug/mL =

meq wt =

Comments:_____

Spike Witness By:_____ Date:_____

Secondary Review By:_____ Date:_____

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ATTACHMENT 13

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Illinois
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N. Billerica,
Massachusetts
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Whippany,
New Jersey
201·428·8181

printed on recycled paper

1.0 APPROVALS

The signature of the following individuals indicates that this Standard Operation Procedure (SOP) is complete and meets the requirements specified in Corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This method is applicable to the measurement of hexavalent chromium in solid wastes.

2.2 This method is applicable to Test Code CR6_02 for hexavalent chromium (Chromium VI) analysis in alkaline digestates of solid wastes.

3.0 SUMMARY OF METHOD

3.1 This method uses an alkaline digestion of waste samples to solubilize both water-insoluble and water-soluble hexavalent chromium compounds. The resulting dissolved hexavalent chromium reacts with diphenylcarbazide at a pH of 0.7 to 1.3 to form a red-violet, water soluble compound which absorbs light at 540 nm.

3.2 The analytical methodology is based on EPA SW-846 Method 7196A, while the digestion procedure is based on SW-846 Method 3060A.

3.3 This method is based on EPA SW-846 Method 7196A chemistry, modified for analysis using a Lachat QuickChem Flow Injection Analyzer. The digestion procedure deviates from SW-846 Method 3060A in the following ways:

3.3.1 The spiking solution is the same as the stock hexavalent chromium (Section 8.2.1.1, 50 mg/L) instead of 1000 mg/L.

3.3.2 The spiking level is fixed at 10 mg/kg instead of twice the concentration of hexavalent chromium found in the unspiked aliquot of sample since the concentration is unknown at the time of sample preparation.

3.3.3 Preparation Blanks must be less than the Practical Quantitation Limit (PQL) instead of less than the MDL or 1/10 of the lowest sample concentration.

3.3.4 The temperature during sample preparation is monitored by immersing the hotplate temperature probe in a sample, not a temperature blank.

4.0 INTERFERENCES

4.1 The chromium reaction with diphenylcarbazide is usually free from interferences, however, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

5.0 SAFETY

5.1 Diphenylcarbazide has no known acute toxicity, although irritation may result from exposure to the skin, respiratory tract and mucous membranes. Consult Material Safety Data Sheets (MSDS) for precautions and proper handling.

5.2 Hexavalent Chromium is a known carcinogen. Avoid direct contact with both the solid powder and solutions of potassium dichromate ($K_2Cr_2O_7$).

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers: Containers may be either glass or plastic, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample Collection: Samples are collected into plastic or glass containers. Samples are cooled and maintained at 4° C and shipped to the laboratory for analysis.

6.3 Sample Preservation: Sample preservation is not necessary. Samples should be refrigerated at 4° C until analysis.

6.4 Holding Times: The holding time for hexavalent chromium is 24 hours from sampling and is applied to the soil matrix as well as the water matrix. If the holding time is exceeded, a Project Status Report (PSR) is completed; informing the project manager that the holding time has been exceeded and that a resampling should be scheduled.

7.0 APPARATUS AND MATERIALS

7.1 Lachat QuikChem Flow Injection Analyzer.

7.2 Programmable Hotplate, with temperature probe.

7.3 250 mL beakers.

7.4 100 mL, 500, 1000 volumetric flasks.

7.5 Short stem funnels.

7.6 pH meter.

7.7 50 mL graduated cylinder.

7.8 Analytical Balance, 0.1 mg sensitivity.

7.9 Watch glasses.

7.10 Filtration apparatus.

7.11 0.45 μ membrane filters.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent water, 16.6 megohm-cm or greater, deionized.

8.1.2 Diphenylcarbizide Color Reagent: In a 1.0 liter volumetric flask, dissolve 0.40 grams s-diphenylcarbazide ($C_{13}H_{14}N_4O$) in 200 mL 99% isopropanol. Add 720 mL reagent water, then 80 mL concentrated sulfuric acid and dilute to volume.

8.1.3 Digestion solution: Dissolve 20.0 grams NaOH and 30.0 grams sodium carbonate (Na_2CO_3) in reagent water and dilute to 1000 mL in a volumetric flask. Store the solution in a tightly capped polyethylene bottle and prepare fresh monthly. The pH of the solution must be checked before usage. The pH must be $\geq$ 11.5 or the solution should be discarded.

8.1.4 Diphenylcarbizide-free Color Reagent: In a 1.0 liter volumetric flask, add 200 mL 99% isopropanol. Add 720 mL reagent water, followed by 80 mL concentrated sulfuric acid and dilute to volume.

8.1.5 Magnesium Chloride: $MgCl_2$ (anhydrous), analytical reagent grade.

8.1.6 1.0 M Phosphate Buffer at pH 7 Preparation:

8.1.6.1 K_2HPO_4, analytical reagent grade.

8.1.6.2 KH_2PO_4, analytical reagent grade.

8.1.6.3 Dissolve 87.09 g K_2HPO_4 and 68.04 g KH_2PO_4 into 700 mL of reagent water. Transfer the solution to a 1 liter volumetric flask and dilute to volume.

8.1.7 Lead Chromate, analytical reagent grade.

8.1.8 Reagent receipt and preparation is documented in the Reagent Preparation and Receiving Logbook (Attachment I).

8.2 Standard Preparation

8.2.1 Calibration Standard Preparation

8.2.1.1 Stock Potassium Dichromate solution (50 mg/L as Cr^{6+}): Dissolve 141.4 mg dried potassium dichromate ($K_2Cr_2O_7$) in deionized water and dilute to 1.0 L.

8.2.1.2 Intermediate Potassium Dichromate solution (4.0 mg/L as Cr^{6+}): Dilute 8.0 mL of the stock solution (Section 8.2.1.1) to 100 mL.

8.2.1.3 Prepare the calibration standards by serial dilution using the following table as a reference.

Standard Concentration	Dilution Std., Volume	Final Volume (mL)
0.40 mg/L	4.0 mg/L, 10.0 mL	100
0.20 mg/L	4.0 mg/L, 5.0 mL	100
0.10 mg/L	0.40 mg/L, 25.0 mL	100
0.04 mg/L	0.40 mg/L, 10.0 mL	100
0.02 mg/L	0.40 mg/L, 5.0 mL	100
0.01 mg/L	0.10 mg/L, 10.0 mL	100

8.2.2 Initial and Continuing Calibration Verification Standard Preparation

8.2.2.1 Initial and Continuing Calibration Verification Standard (ICV, CCV, respectively), 0.25 mg/L as Cr^{6+}: Dilute 10.0 mL of 12.5 mg/L Hach Standard (Purchased from Hach Co., Loveland Co. Catalog # 14256-10) to 500 mL.

8.2.3 All standard receipt and preparation is documented in the Standards Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 The working range of the calibration is 0.01 to 0.40 mg/L as Cr^{6+}.

9.2 Instrument Startup:

9.2.1 Switch the power strip located behind the system computer to the "on" position. When the computer has completed the self-diagnostics, the CPU monitor will display the main menu with the METHODS category illuminated.

9.2.2 Install the Hexavalent chromium chemistry manifold onto the Lachat module, making proper connections to reagent lines as shown in the diagram in the Lachat method manual.

9.2.3 Place all reagent lines into proper reagent containers and adjust the tension levers on the peristaltic pump until the first "click" is heard. While the system is pumping reagents through the manifold, proceed with METHOD DOWNLOADING.

9.3 Method Downloading

9.3.1 To download the chemistry program to the system computer, observe the "METHODS" category illuminated at the CPU monitor, press [ENTER].

9.3.2 The monitor will display "Download Method" and "Method Definition". Using the arrow keys, highlight "Download Method" and press [ENTER].

9.3.3 A menu will appear listing pre-programmed methods in the system. Move the cursor until "HEXAVALENT CHROMIUM" is highlighted, then press ENTER.

9.3.4 The method will now start downloading the chemistry method to the computer.

9.4 Calibration

9.4.1 To set up the standard curve on the autosampler, obtain a vial for each standard beginning with the 0.40 mg/L standard. Fill the vial and place it in the standards rack in the front left spot. Continue with the other standards, in order of decreasing concentration.

9.4.2 The computer monitor will display "Edit". Using the arrow keys, highlight "Submit" and "Calibrate Now" then hit [ENTER]. The screen will give the prompt "Should a calibration be started now?", press "Y" for yes.

9.4.3 The Lachat will begin calibrating. When the calibration is complete the system will indicate whether the pass/fail criteria has been met by displaying "calibration failed!" or "calibration approved".

Note: Analysis cannot proceed until the calibration is approved.

9.5 Acceptance criteria for the calibration requires the linear regression correlation coefficient of the calibration standards to be greater than 0.9975.

9.6 Calculations: Analyte concentrations in samples are calculated by Lachat software which is based on linear regression.

10.0 QUALITY CONTROL

10.1 Method detection limits are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET which is based on the 40 CFR, Part 136, Appendix B definition of method detection limit.

10.2 The practical quantitation limit for this method is 0.5 mg/kg.

10.3 The Lachat software performs a diagnostics check on the autosampler, switching valves and the detector after the initial powering-up of the system computer.

10.4 Soluble Pre-Digestion Matrix Spikes: Pipet 2.0 mL stock potassium dichromate solution (Section 8.2.1.1, 50 mg/L) to 2.5 grams of sample, and prepare as in Section 11.2. This comprises a matrix spike of 40 mg/kg at analysis. Matrix spikes are analyzed with every 20 samples or less, per batch.

10.5 Insoluble Pre-Digestion Matrix Spikes: Add 10 - 20 mg of lead chromate (Section 8.1.7) to 2.5 grams of sample and prepare as in Section 11.2.

10.6 Post-Digestion Matrix Spike: Pipet 2.0 mL stock potassium dichromate solution (Section 8.2.1.1, 50 mg/L) to a previously digested sample. This comprises a matrix spike of 40 mg/kg at analysis. Matrix spikes are analyzed with every 20 samples or less, per batch.

10.6.1 If the post-digestion spike does not meet the acceptance criteria defined in Section 13.1, the sample should be reanalyzed at a 2X dilution. This should be repeated at additional dilutions until the post-digestion spike recovery criteria is met.

10.7 Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), (Section 8.2.2) represent QC check samples for this method. ICVs are analyzed at the onset of analysis and CCVs are analyzed every 10 samples.

10.8 Initial and Continuing Calibration Blanks (ICB and CCB, respectively) consist of digestion solution (Section 8.1.3). ICBs are analyzed at the onset of analysis and CCBs are analyzed every 10 samples.

10.9 Preparation Blanks consist of digestion solution (Section 8.1.3) that is prepared as in Section 11.2. Preparation Blanks are analyzed with every 20 samples or less, per batch.

10.10 Duplicate samples are analyzed with every 20 samples or less, per batch.

10.11 Surrogates are not applicable to this method.

10.12 Internal standards are not applicable to this method.

10.13 The Laboratory Control Sample (LCS) is prepared by spiking 50 mL of the digestion solution (Section 8.1.3) with 0.4 mL of ICV Calibration Standard (Section 8.2.2.1). LCSs are analyzed with every 20 samples or less, per batch.

10.14 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
ICV/ICB	Onset of every analysis
Preparation Blank	Every 20 samples, per batch
Duplicate sample	Every 20 samples, per batch
Soluble Pre-Digestion Matrix Spike	Every 20 samples, per batch
Insoluble Pre-Digestion Matrix Spike	Every 20 samples, per batch
Post Digestion Matrix Spike	Every 20 samples, per batch
QC Check Samples (CCV/CCB)	Every 10 samples, per analysis
Laboratory Control Sample	Every 20 samples, per batch

DEFINITIONS:
Batch: Every 20 samples or less leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions

11.1.1 See Section 9.0 for specific procedures for instrument startup and calibration.

11.2 Sample Preparation

11.2.1 The methodology for the preparation of soils for hexavalent chromium analysis is based on an Alkaline Digestion method, SW-846 Method 3060A. This procedure is reduced by a factor of 10 for use with the Lachat Flow Injection Analyzer.

11.2.2 Homogenize the sample in accordance with the SOP for Homogenization and Percent Solids, IEA Doc# QAS01400.NC.

11.2.3 Place 2.5 grams of sample into a 250 mL beaker and record the weight in the Lachat Runlog (Attachment III).

11.2.4 Add 50 mL of digestion solution (Section 8.1.3) to each sample. Add 392.18 mg of $MgCl_2$ (Section 8.1.5) and 0.5 mL of 1.0 M phosphate buffer (Section 8.1.6). Stir with a stir bar for at least 5 minutes unheated. Cover the beaker with a watch glass and continue to stir until the 60 minute digestion is complete (Section 11.2.9).

11.2.5 Press the [ON/OFF] key on the programmable hotplate to turn the power on and set the stirrer speed to 250 rpm by pressing [ENT], [3], [2][5][0], [ENT].

11.2.6 Place the temperature probe in a sample beaker as prepared in Section 11.2.4 making sure that the tip of the probe is completely submersed.

11.2.7 Set the target probe temperature to 95 degrees C by pressing [ENT], [2], [9][5], [ENT]. At this point the display will flash the current temperature and the target temperature repeatedly.

11.2.8 When the current temperature reaches 92 degrees C set the timer to one hour. Do not allow the sample to go to dryness as hexavalent chromium may be lost.

11.2.9 When one hour has expired, stop the heater by pressing [ENT], [2], [2][0], [ENT].

11.2.10 When the current temperature reaches 50 degrees C, turn the power to the hotplate off.

11.2.11 Up to nine sample may be digested on the programmable hotplate at one time, using one sample with the temperature probe.

11.2.12 Gradually cool each solution to room temperature and transfer it quantitatively to the filtration apparatus with reagent water rinses and filter through a 0.45 μ membrane filter.

11.2.13 Rinse the inside of the filter flask and filter pad with reagent water and transfer the filtrate and the rinses to a clean 250 mL beaker.

11.2.14 Place a stir bar in the filtrate beaker and, with constant stirring, slowly add nitric acid dropwise until the pH is 7.5 $\pm$ 0.5. Monitor the pH with a pH meter. If the pH drops below 7.0 discard the solution and digest again.

11.2.15 Remove and rinse the stir bar. Collect the rinsate in the filtrate beaker and transfer the contents of the beaker to a 100 mL volumetric flask and dilute to volume in reagent water. The sample is now ready for analysis.

11.2.16 Repeat Sections 11.2.1 - 11.2.15 until all samples including QC samples have been prepared.

11.3 Sample Analysis

11.3.1 All samples are analyzed initially at a 5x dilution.

11.3.2 After obtaining an approved calibration (Section 9.0), press the [Esc] key until the "Sample Tray" program menu appears. Use the arrow keys to highlight "Edit" then press [ENTER].

11.3.3 A sub menu will appear with "Identification" highlighted. Press [ENTER].

11.3.4 Proceed to enter the sample ID's in the exact order that they are arranged in the sample tray. After the last sample has been entered, type two periods " .. " as the next sample entry; this indicates the end of the tray.

11.3.5 Press the [ESC] key to back out to the "Sample tray" menu once again. Using the arrow keys, highlight "SUBMIT" and then press [ENTER].

11.3.6 The display will prompt the operator to submit the tray for analysis.

Note: Real time display of sample results may be accessed by pressing the [CTRL], [SHIFT], and [1] keys simultaneously. To return to the program menu, simply press [CTRL], [SHIFT], and [2] simultaneously. DO NOT use the arrow keys to scroll the screen in this mode; it will cause the computer to lock up. This will require the system to be shut down and the method downloading and calibration to be executed again.

11.4 After all analyses are complete, switch the color reagent line to the diphenylcarbizide free reagent (Section 8.1.4) and rinse for 5 minutes.

11.5 The analysis run is then resubmitted exactly as it was performed previously. The absorbance from this run is subtracted from the color reagent run to correct for nonspecific absorbance for all data points. This should be noted on the final report to the client. Both corrected and uncorrected data should be maintained on file.

11.6 All analyses including calibration, samples, and QC Samples are documented in the Lachat Runlog (Attachment III).

12.0 CALCULATIONS

12.1 ICV,and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{Theor. mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{\text{(Spiked sample + sample conc., mg/kg) - (sample conc., mg/kg)}}{\text{Spike Added (mg/kg)}} \times 100 = \%\ \text{Recovery}$$

12.3 Relative Percent Difference:

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

13.0 ACCEPTANCE CRITERIA

13.1

Blanks	< 0.5 mg/kg (PQL)
ICV, CCV	85% - 115% Recovery
Pre-digestion MSs	75% - 125% Recovery
Post-Digestion MS	85% - 115% Recovery
Duplicates	± 20% RPD or ± MDL if sample conc. < 5 x MDL
LCS	85% - 115% Recovery

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: 0.5 mg/kg as Cr^{6+}
Units of Measure: mg/kg
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL

QC information included in report:
Level 1 QC: Blank values, sampling COC.
Level 2 QC: Blank values, Duplicate data (%RPD), sampling COC.
Level 3 QC: Blank values, Duplicate data (%RPD), sampling and internal COC, matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 Corporate SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.2 SOP for Soil Homogenization and Percent Solids, IEA Doc#QAS01400.NC.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Wastes, 3rd Edition, Method 7196A, July 1992 Revision.

16.2 Test Methods for Evaluating Solid Wastes, 3rd Edition, Method 3060A, January 1995 Revision.

16.3 Lachat QuikChem Flow Injection Analyzer Method #10-124-13-1-A.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 8.1.1 revised for correct units. Sections 8.1.7 and 11.4 added. COCs added to Level 2 and Level 3 report QC. Sections 8.2 and 11.5 added. Logbook reference added to Sections 9.1 and 11.2.1. SOP reference added in Section 10.1.

17.2 Sections 3.3.3, 7.8-7.12 and 11.3.1 added. Sections 3.2, 3.3 and 16.1 revised to the current reference method.Section 8.0 revised for clarification including the addition of sections 8.1.5, 8.1.6 and 8.1.7. Section 10.0 revised for clarification including the addition of Section 10.4-10.6 and 10.13-10.14. Section 11.0 revised for clarification including the addition of Sections 11.2.2 and 11.2.5-11.2.16. Section 11.2.4 revised to 50 mL of digestion solution, the addition of magnesium chloride and the addition of phosphate buffer for method compliance. Sections 13.1 and 14.1 revised to reflect the correct PQL. Pre-digestion spike, post-digestion spike and LCS acceptance criteria added in Section 13.1. (05/10/96)

17.3 Section 10.6.1 added. (12/10/96)

Attachment I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
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Secondary Review By:_____ Date:_____

IEA, Inc. Form# CVL01200.NC

IEA Logbook# CV17

Attachment II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

:ondary Review By:_____ Date:_____

Attachment III

Lachat Runlog

Instrument-#1

Analyst: _____

Preparation Log: _____

Filename: _____

Sample ID.	Sample Amount (g,ml)	Leachate/Dig Volume		Dilution		DF	Analysis mg/l,ug/l	Result mg/l,mg/kg	Comments
		g/ml	Final Volume	Vi	Vf				

Secondary Review By:_____ Date:_____

IEA, Inc. Form# CVF00200.NC

Analysis Date: _____

Chemistry: _____

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director _____

Quality Assurance Manager _____

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This procedure is applicable to the measurement of total and dissolved sulfides in drinking surface and saline waters, domestic and industrial wastes as well as soil sample leachates. Excess iodine is added to a sample which may or may not have been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is backtitrated with sodium thiosulfate or phenylarsine oxide. This procedure is suitable for the measurement of sulfide in concentrations above 1 mg/L.

2.2 Acid insoluble sulfides are not measured by this test. (Copper sulfide is the only common sulfide in this class.

2.3 This procedure is applicable to Test Codes S_01 & S_02.

3.0 SUMMARY OF METHOD

3.1 200 mL or an aliquot of sample diluted to 200 mL is placed in a 250 mL erlenmeyer flask. 2 mL of 6N HCl and 10 mL of 0.025N iodine solution are then added to the flask. The solution in the flask is titrated with sodium thiosulfate solution until a pale yellow color is obtained. 6-8 drops of starch indicator is then added to the erlenmeyer flask resulting in a dark blue solution. The solution in the erlenmeyer is backtitrate with sodium thiosulfate solution until the blue color is no longer visible. Initial and final volumes of titrant are recorded to determine the mg/L of sulfide present.

3.2 This procedure is based on EPA Method 376.1.

3.3 This SOP deviates from EPA Method 376.1 in the following manner:

3.3.1 This SOP is modified to accommodate the analysis of soils.

4.0 INTERFERENCES

4.1 Reduced sulfur compounds, such as sulfite, thiosulfate and hydrosulfite which decompose in acid may yield erratic results. Also, volatile iodine-consuming substances will give high results.

4.2 Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.

4.3 If the sample is not preserved with zinc acetate, the analysis must be started immediately. Similarly, the measurement of dissolved sulfides must be also begin immediately.

5.0 SAFETY

5.1 The use of sodium thiosulfate solution, and 6N HCL acid solution may cause skin irritation. Safety glasses, lab coat, and latex gloves are necessary to prevent contact with these solutions.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers must be glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample collection: Samples are collected with zinc acetate as a preservative and stored at 4 °C until analysis. Samples are shipped to the laboratory at 4 °C and stored at 4 °C until analysis.

6.3 Sample Preservation

6.3.1 Water samples are preserved with 2 mL zinc acetate and NaOH to a pH > 9.

6.3.2 Soil samples are cooled to 4 °C until analysis.

6.4 The holding time for sulfide analysis is 7 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 25 mL buret.

7.2 250 mL Erlenmeyer flasks.

7.3 10 mL pipet.

7.4 250 mL graduated cylinder.

7.5 Magnetic stirrer, stir bar.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent water, 16.6 megohm or higher, deionized.

8.1.2 Concentrated Hydrochloric Acid, purchased from a commercial vendor.

8.1.2.1 6N HCl: Carefully dilute 125 mL of concentrated HCl (Section 8.1.2) to 250 mL with reagent water (Section 8.1.1).

8.1.3 Iodine solution, 1N, purchased from a commercial vendor.

8.1.3.1 0.025N iodine solution: Dilute 25 mL 1N iodine solution (Section 8.1.3) to 1000 mL with reagent water (Section 8.1.1).

8.1.4 Soluble starch, purchased from a commercial vendor.

8.1.5 Salicylic acid, purchased from a commercial vendor.

8.1.6 Starch indicator solution: Dissolve 5 g starch (Section 8.1.4) and 0.5 g salicylic acid into 200 mL of hot reagent water with constant stirring until dissolved. Dilute to a final volume of 250 mL with reagent water (Section 8.1.1).

8.1.7 All reagent receipt and preparation is documented in the Reagent Receipt and Preparation Logbook (Attachment I).

8.2 Standard solutions

8.2.1 Sodium Thiosulfate solution, 0.025N NaS_2O_3 certified lot analysis from Fisher Scientific Co., (Cat. no. SS370.1).

8.2.2 All standard receipt and preparation is documented in the Standard Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 Calibration is not applicable.

9.2 The working range of this method is 1 mg/L and above. Samples requiring in excess of 50 mL of standard titrant are diluted within range.

10.0 QUALITY CONTROL

10.1 Method Detection Limits (MDLs) are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

10.2 The practical quantitation limit (PQL) is 1 mg/L for water samples and 1 mg/kg for soil samples.

10.3 Matrix spikes are not practical for this method.

10.4 QC check samples consist of Initial and Continuing Blanks. Blanks are performed at the onset of analysis and after every ten samples.

10.4.1 Blanks are prepared by aliquoting 200 mL of reagent water and analyzing as other samples.

10.5 Surrogates are not applicable to this method.

10.6 Duplicate samples are analyzed every 10 samples.

10.7 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Blank	Onset of every analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Duplicate Samples	Every 10 samples, per analysis

10.7.1 Definitions

Batch: Every 10 samples, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Water Samples

11.1.1 Rinse a clean 25 mL buret three times with titrant (sodium thiosulfate, Section 8.2.1). Fill the buret, making sure that no air bubbles are present.

11.1.2 Add 200 mL of sample to a 250 mL erlenmeyer flask. If the sample appears to be turbid or cloudy, a smaller aliquot may be diluted to 200 mL. Record the initial volume of sample used in the Titrimetric Analysis Logbook (Attachment III).

11.1.3 Add 10.0 mL of 0.025N iodine solution (Section 8.1.3.1) and 2 mL of 6N HCl (Section 8.1.2.1).

11.1.4 Record the initial volume of titrant in the Titrimetric Analysis Logbook.

11.1.5 Titrate the solution in the erlenmeyer with sodium thiosulfate until a pale yellow color is achieved. Record the final volume of titrant in the Titrimetric Analysis Logbook.

11.1.6 Add 6-8 drops of starch indicator (Section 8.1.6). The solution in the erlenmeyer should be dark blue.

11.1.7 Record the initial volume of titrant in the Titrimetric Analysis Logbook.

11.1.8 Titrate the solution in the erlenmeyer with sodium thiosulfate until the solution is clear. Record the final volume of titrant in the Titrimetric Analysis Logbook.

11.2 Soil Samples

11.2.1 Weigh 25 g of soil sample into an erlenmeyer flask and add 250 mL of reagent water. Let the erlenmeyer stand for 20 minutes. Record the weight of sample leached in the Titrimetric Analysis Logbook.

11.2.2 Decant the supernatant and centrifuge. Pour off 200 mL of the centrifuged solution into a 250 mL erlenmeyer flask and titrate as in Sections 11.1.3 - 11.1.8.

11.3 Samples Containing Precipitate

11.3.1 Samples with precipitate should be titrated directly in the original bottle as in Sections 11.1.3 - 11.1.8.

11.3.2 When the titration is complete, transfer the contents of the sample bottle to a graduated cylinder to determine the original sample volume (subtract the volume of titrant added).

12.0 CALCULATIONS

12.1 Water Samples

$$\text{mg-sulfide/L} = \frac{[(ab)-(cd)] \times 16000}{\text{volume of sample (mL)}}$$

a = mL of iodine solution (10 mL)
b = Normality of iodine solution (0.025N)
c = mL of sodium thiosulfate used
d = Normality of sodium thiosulfate (0.025N)

12.2 Soil Samples

$$\text{mg sulfide/kg} = \frac{[(ab)-(cd)] \times 16000 \times 250 \text{ mL}}{\text{wt of sample leached (g)}}$$

a = mL of iodine solution (10 mL)
b = Normality of iodine solution (0.025N)
c = mL of sodium thiosulfate used
d = Normality of sodium thiosulfate (0.025N)

12.3 Relative Percent Difference

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

13.0 ACCEPTANCE CRITERIA

13.1 ICB, CCB < PQL
DUPLICATES +/- 20% or +/-1 mg if sample conc. <5 x MDL.

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limit: 1.0 mg/L for waters and 1.0 mg/kg for soils
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level I QC: Blank values, Sampling COCs.
Level II QC: Blank values, Duplicate data (%RPD), Sampling COCs.
Level III QC: Blank values, Duplicate data (%RPD) and Sampling and Laboratory COCs.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 Methods for Chemical Analysis of Water and Waste, 1983, EMSL, EPA-600/4-79-020. Method 376.1.

17.0 SUBSTANTIVE REVISIONS

17.1 Original Issue

17.2 Section 8.1.1 revised from 18.0 megohm distilled water to 16.6. Section 11.2.5 added to address precipitated samples. (04/18/95)

17.3 Section 2.0 revised for clarification. Sections 3.3, 6.3.2, 10.4.1, and 12.2 added. Section 8.0 revised for clarification. Duplicate frequency added in Section 10.7. Section 12.1 revised. Sections 10.2 and 14.1 revised to reflect current PQLs for soil and water samples. Section 12.2 added. (08/08/96)

Attachment I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
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Secondary Review By:_____ Date:_____

IEA, Inc. Form# CVL01200.NC

IEA Logbook# CV17

Attachment II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:_____ **Date:**_____

Attachment III

TITRIMETRIC ANALYSIS

Method Name:_____ DATE:_____ Analyst:_____

Daily Standards and Spike Preparation

Standard ID	Stock Lot#	Stock Conc	Stock Vol	Diluent	Final Volume	Final Conc	Std #
ICV/QC Check							
Working Standard							
Spike							

TITRANT:_____ Normality:_____ Reference:_____

Sample ID	mLs Sample	Titrant V_I	Titrant V_F	Titrant V_T	ug/mL	%Rec. / RPD
ICV						
ICB						

CALCULATIONS:

$$RPD = \frac{|(\text{Sample} - \text{Sample Dup.})|}{\text{Avg., Sample} + \text{Dup.}} \times 100 \quad \% \text{ Recovery} = \frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spike Concentration}} \times 100$$

Concentration:

ug/mL =

meq wt =

Comments:_____

Spike Witness By:_____ Date:_____

Secondary Review By:_____ Date:_____