APPENDICES

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APPENDIX A

SEDIMENT SAMPLING PLAN (SSP)

SEDIMENT SAMPLING PLAN

Site Name /Project Name:	Removal of the Maxwell Pond Dam/Black Brook Dam (No. 150.07) and restoration of Black Brook
Site Location:	Manchester, New Hampshire
Client:	City of Manchester
Partner Agency:	New Hampshire Department of Environmental Services (DES)
Prepared for:	Deborah S. Loiselle, River Restoration Coordinator (DES) Lori S. Siegel, PhD.,P.E., Ecological Risk Assessor (DES)
Prepared by:	Jeffrey Tucker, Dubois & King, Inc.
Date:	January 4, 2008

1.0 OVERVIEW

The overall sediment investigation is designed to identify management approaches for the sediments within the Maxwell Pond impoundment. Under the alternative of removing the dam, accumulated sediment would erode in part and be transported into the Merrimack River downstream of the dam. The primary purpose of this investigation is to analyze the contaminant content of the sediment that has accumulated in the impoundment.

2.0 SAMPLING PLAN

Sampling Locations

Two samples are to be collected within the impoundment (one adjacent to the dam and one in the upper reaches of the impoundment). A third and fourth sample will be collected downstream of the impoundment and upstream of the effects of the impoundment respectively. Approximate sampling locations are shown on Figure 1. Sampling locations will be determined based upon (a) the approximate location of the former stream channel, (b) the accumulated sediment thickness, and (c) substrate availability within the upstream and downstream channels. Sampling and analysis procedures will follow NHDES guidelines ("Evaluation of Sediment Quality for Dam Removal" and "Evaluation of Sediment Quality Guidance Document").

The approximate location of the stream channel will be ascertained from the bathymetry map prepared by The New Hampshire Department of Environmental Services and Trout Unlimited. The thickness of accumulated sediment at sampling locations will be estimated based on sediment depth probes completed previously by NHDES. Approximate sediment depth at the points of sampling will be field collected via manual probing by Dubois & King personnel with assistance provided by the NHDES and the City of Manchester. Sediment depths greater than 12-feet will be reported as such. Sampling stations will be located at the following locations (depicted in Figures 1 and 2 below and 3 through 6 at the end of this SSP):

MP-S1: Upstream of the confluence of Black Brook and Maxwell Pond (if possible)
MP-S2: Within Maxwell Pond situated in the historical Black Brook stream channel
MP-S3: Immediately upstream of the Maxwell Pond Dam in the impoundment
MP-S4: Downstream of the Maxwell Pond Dam above the backwater influence of the Merrimack River

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Figure 1: Maxwell Pond Locus Map With Sediment Sampling Locations

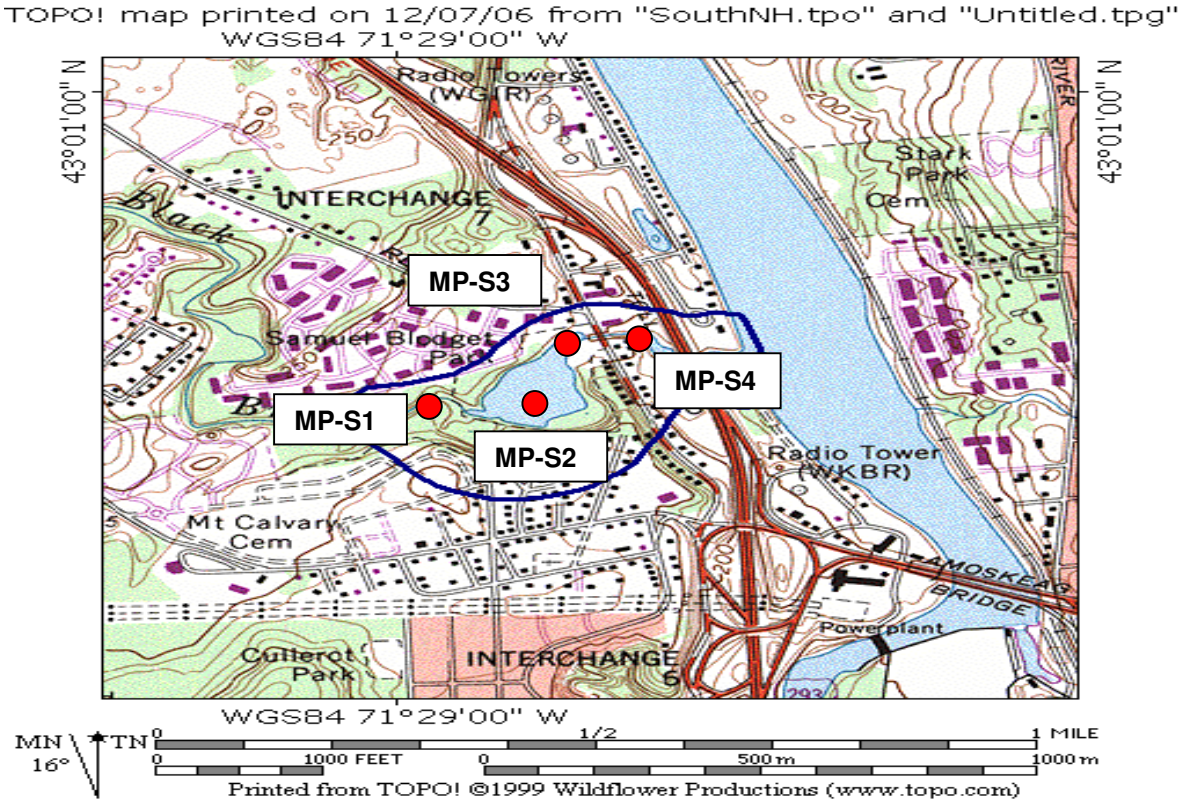
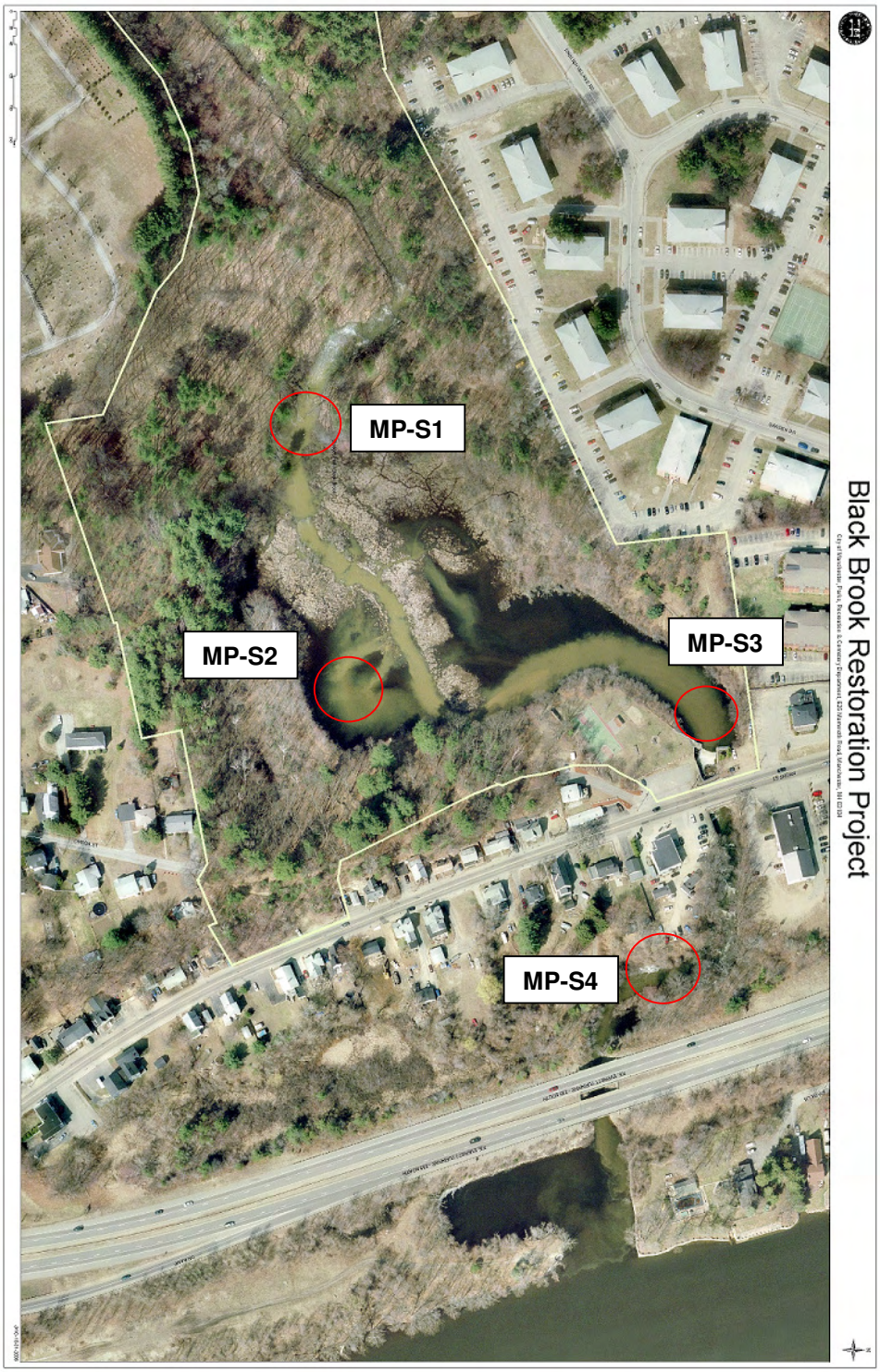


Figure 2: Maxwell Pond Impoundment & Black Brook Aerial Map With Proposed Sampling Areas



Sampling Equipment

Appropriately cleaned sample bottles will be provided by the laboratory. All bottles will be sterilized/cleaned at the Endyne, Inc. Laboratory, sealed and kept in clean bags for transportation to and from site. Nitrile gloves will be used in collecting the samples to avoid the possibility of phthalate contamination from handling samples with plastic gloves.

A hand held hollow-stem auger will be used that will allow ¾" x 24" plastic retaining cylinder cores to retrieve field sediment samples . If possible, ¾" x 48" plastic retaining cylinder cores will hand pushed into the sediment to retrieve the sediment samples. Sampling tools will depend on the consistency and thickness of the sediment. The goal is to collect a representative sample of the sediment column that accumulated at each location since the dam was built. Hollow-stem auger drilling uses continuous- flight augers that mechanically excavate drilled materials from the hole. These augers are built with a hollow shaft (plugged while augering) to allow access to the bottom of the hole without withdrawing the auger. The augers act as temporary casing to facilitate the sampling of sediment. All instrumentation and equipment must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the SSP.

All equipment used to collect and handle samples must be cleaned and all parts examined to ensure proper functioning before going into the field. Backup equipment and sampling gear should be available. To ensure sediment samples are not contaminated from one sample location to the next, re-usable sampling equipment will be decontaminated following the below guidelines:

1. Wash with a scrub brush and a non-phosphate detergent with water
2. Rinse with clean water
3. Rinse again with deionized water

To further minimize cross-contamination leading to false detections, upstream locations will be sampled before downstream locations.

Sample Collection

Each sample will be collected by DuBois & King in accordance with the protocols and guidance provided by DES (Evaluation of Sediment Quality Guidance Document, NHDES-WD-04-9) and EPA (EPA-823-B-01-002, 2001). Samples will be collected from one borehole at each of the four (4) sample locations. Sediment will be recovered in 24" lengths from the pond bottom surface to refusal or limits of the sampling equipment. It is anticipated that a maximum depth of twelve (12)-feet from the water surface will be achievable.

The recovered cores from each sample location will be examined for evidence of stratification based on visual variation in color and/or sediment size. The recovered material will be composited into a single sample for laboratory analysis. Compositing will be done in a homogenization container prior to transferring to the laboratory containers. If however, a sample cannot be collected at a proposed location (such as upstream and downstream of the pond where there is bedrock present) the recovered material of the remaining locations will be separated into multiple samples based on stratification. A total of four samples will be submitted for laboratory analysis. Samples for the VOC testing will not be composited but will be transferred directly into the laboratory containers from the sampling core.

Field notes will be taken by field personnel to provide a detailed description of the sampling event utilizing the Sediment Sampling Field Notes form attached in Appendix A. Information for each sediment core will be recorded on the Field Log Form attached in Appendix A.

Samples will be placed in coolers with ice. The cooler will be transported to the laboratory, together with a completed Chain-of-Custody form and instruction for the laboratory for compositing of the subsamples for each area. Subsamples from each area will be labeled separately, e.g., MP-S1a, MP-S1b, MP-S1c. Samples

will be analyzed by Endyne, Inc. Laboratory Services. Standard turn-around time for the laboratory is 2 weeks.

3.0 ANALYSIS

A State-certified laboratory (Endyne, Inc. Laboratory Services) will complete the analytical work for the following parameters:

- VOCs by USEPA Method 8260B
- SVOCs by USEPA Method 8270C
- Polynuclear aromatic hydrocarbons (PAHs) by USEPA Method 8270C
- Pesticides by USEPA Method 8081A
- Polychlorinated byphenyls (PCBs) by USEPA Method 8082
- Selected metals [arsenic, barium, cadmium, chromium (total), copper, lead, mercury, nickel, zinc by USEPA Methods 6010B and 7174 (mercury only)]
- Total organic carbon
- Grain size distribution via sieve and hydrometer by American Society for Testing and Materials (ASTM) method D-422, or a comparable method

Parameter	CAS. No.	Method	MDL[5]	Reporting Limits[5]	Sediment Screening Thresholds[5,6]	
					Threshold Effect Concentration (TEC)	Probable Effect Concentration (PEC)
VOCs[10]		8260B[9]	0.51 ug/kg – 19.78 ug/kg	10 ug/kg - 200 ug/kg	See Note 6	See Note 6
SVOCs		8270C	5.34 ug/kg- 64.83 ug/kg	0 ug/kg - 2,000 ug/kg		
PAHs (All SVOCs)		8270c-SIM	7.31 ug/kg- 25.01 ug/kg	80 ug/kg		
Anthracene	120127		21.58 ug/kg	80.0 ug/kg	57.2 ug/kg[4]	845 ug/kg[4]
Benz(a)anthracene	56553		13.51 ug/kg	80.0 ug/kg	108 ug/kg[4]	1,050 ug/kg[4]
Benzo(g,h,i)perylene	191242		25.01 ug/kg	80 ug/kg	290 ug/kg[2]	6,300 ug/kg[2]
Benzo(a)pyrene	50328		19.82 ug/kg	80.0 ug/kg	150 ug/kg[4]	1,450 ug/kg[4]
Chysene	218019		18.37 ug/kg	80.0 ug/kg	166 ug/kg[4]	1,290 ug/kg[4]
Dibenz(a.h)anthracene	53703		17.37 ug/kg	80.0 ug/kg	33.0 ug/kg[4]	330 ug/kg[2]
Fluoranthene	206440		19.82 ug/kg	80.0 ug/kg	423 ug/kg[4]	2,230 ug/kg[4]
Fluorene	86737		11.77 ug/kg	80.0 ug/kg	77.4 ug/kg[4]	536 ug/kg[4]
Indeno(1,2,3-cd)pyrene	193395		14.75 ug/kg	80 ug/kg	78 ug/kg[2]	836.66 ug/kg[2]
Naphthalene	91203		9.77 ug/kg	80.0 ug/kg	176 ug/kg[4]	561 ug/kg[4]
Phenanthrene	85018		9.50 ug/kg	80.0 ug/kg	204 ug/kg[4]	1,170 ug/kg[4]
Pyrene	129000		16.01 ug/kg	80.0 ug/kg	195 ug/kg[4]	1,520 ug/kg[4]
PAH, Total	1000				1,610 ug/kg[4]	22,800 ug/kg[4]

Parameter	CAS. No.	Method	MDL[5]	Reporting Limits[5]	Sediment Screening Thresholds[5,6]	
					Threshold Effect Concentration (TEC)	Probable Effect Concentration (PEC)
Pesticides (All SVOCs)		8081A	0.69 ug/kg – 2.34 ug/kg	<10 ug/kg		
Chlordane	57749		2.34 ug/kg	<20 ug/kg	3.24 ug/kg[4]	17.6 ug/kg[4]
Dieldrin	60571		1.21 ug/kg	<10 ug/kg	1.90 ug/kg[4]	61.8 ug/kg[4]
Sum DDD	72548		1.07 ug/kg	<10 ug/kg	4.88 ug/kg[4]	28.0 ug/kg[4]
Sum DDE	72559		1.38 ug/kg	<10 ug/kg	3.16 ug/kg[4]	31.3 ug/kg[4]
Sum DDT	50293		0.98 ug/kg	<10 ug/kg	4.16 ug/kg[4]	62.9 ug/kg[4]
Total DDTs					5.28 ug/kg[4]	572 ug/kg[4]
Endrin	72208		0.69 ug/kg	<10 ug/kg	2.22 ug/kg[4]	207 ug/kg[4]
Heptachlor epoxide	1024573		1.50 ug/kg	<10 ug/kg	2.47 ug/kg[4]	16.0 ug/kg[4]
Lindane (gamma-BHC)	58899		2.09 ug/kg	<10 ug/kg	2.37 ug/kg[4]	4.99 ug/kg[4]
PCB Aroclors		8082	See App. B	<50 ug/kg		
Total PCB	1336363		See App. B	See App. B	59.8 ug/kg[4]	676 ug/kg[4]
TOC		9060		100 ug/g[7]	NA	NA
Sieve with hydrometer		ASTM D422		#4, 10, 40, 200 and silt and clay		NA
Metals		6010B/ 7471A	0.144 mg/kg – 1.09 mh/kg	0.50 mg/kg- 3.00 mg/kg		
Arsenic	22569728		0.676 mg/kg	2.00 mg/kg	9.79 mg/kg[4]	33 mg/kg[4]
Barium[8]	7440393		0.373 mg/kg	2.00 mg/kg		48 mg/kg [1]
Cadmium	7440439		0.150 mg/kg	0.50 mg/kg	0.99 mg/kg[4]	4.98 mg/kg[4]
Chromium	1308141		0.257 mg/kg	1.00 mg/kg	43.4 mg/kg [4]	111 mg/kg [4]
Copper	7440508		0.235 mg/kg	1.00 mg/kg	31.6 mg/kg [4]	149 mg/kg [4]
Lead	7439921		0.468 mg/kg	2.00 mg/kg	35.8 mg/kg [4]	128 mg/kg [4]
Nickel	7440020		0.334 mg/kg	1.50 mg/kg	22.7 mg/kg [4]	48.6 mg/kg [4]
Zinc	7440666		0.373 mg/kg	1.50 mg/kg	121 mg/kg [4]	459 mg/kg [4]
Mercury	7439976		0.300 mg/kg	0.60 mg/kg	0.18 mg/kg [4]	1.06 mg/kg [4]

Notes:
1. NOAA 1999 – SQuiRT Tables (NOAA Hazmat Report 99-1).
2. Oak Ridge National Laboratory 1997 – Toxicological Benchmarks (ORNL ES/ER/TM-95/R4)
3. US Environmental Protection Agency 1996 – Ecotox Thresholds (USEPA EPA 540/F-95/038)
4. MacDonald et al., – Archives of Environmental Contamination and Toxicology Vol 39, No. 1: p. 20-31 (June, 2000).
5. For each contaminant, an appropriately conservative Threshold Effect Concentration (TEC) and Probable Effect Concentration (PEC) were researched from available guidelines for each contaminant of concern (Notes 1-4 listed above). TEC values are screening thresholds below which adverse effects are unlikely. PEC values are screening thresholds above which adverse effects are likely. For freshwater environments, although consensus-based (CB) TEC and PEC values (MacDonald et al., 2000) are not the most conservative, they do have the most statistical justification (NHDES-WD-04-9). Values from Sources (Notes) 1-3 were utilized when a value could not be found in MacDonald et al., 2000. Only compounds with a known TEC and PEC are shown even though there may be numerous other compounds tested for by laboratory (SVOCS, PAHs, Pesticides, PCBs , etc). See attached Endyne 2007 Quality Control Data in Appendix B for full suite of compounds for each analyte category. Reported as dry weight.
6. For contaminants with no available threshold, such as VOC, a standard will be calculated using the contaminant's surface water standard with appropriate partitioning coefficients. This will be done after receipt of lab results if need be. Reported as dry weight.
7. Endyne, Inc. (Adirondack Environmental Services) TOC SOP data..
8. Barium is a TCLP compound. It is comparable to silver for MDL and RL values.
9. Endyne Inc's SOP for Volatile Organics analysis is method "EPA 8260B". Appendix A of this SOP only references the soil preparation component of the analysis method to be "EPA 5035". The actual analytical method is still EPA 8260B.
10. See sampling procedure described in Volatile Organic Compounds Method EPA 8260/8260B SOP's Appendix A attached in QAPP Appendix G

The list of parameters, reporting limits (RL), and method detection limits (MDL) was provided by the laboratory for the analyses. The laboratory will report the concentration down to the method detection limits (MDL), flagging values between the MDL and the RL and equal to or exceeding the TEC and PEC values.

A duplicate sample is not planned due to the low number of planned samples, and due to the homogeneity of the sediment supply. Specifically, most of the sediment that enters the impoundment is supplied by Black Brook to the west. In addition, coarser-grained sediments are likely trapped by impoundments upstream. The results of the parameters tested for will be compared to the sediment screening thresholds indicated in the table above. See Data Evaluation below.

Data Management

Field data will be recorded on field logs and soil boring logs (see Appendix A). The field and laboratory data will be incorporated into a spreadsheet for the project. Information such as sample ID, date and time of collection, and other field data will be recorded. This step will create a record for each sample prior to receipt of the analytical data from the laboratory and will be the initial step of setting up a sample tracking system. The sample tracking system will allow the status of each sample to be identified during the data generation process. Additionally, full laboratory data packages will be supplied for the PAHs, metals and other analyses in a scanned electronic media (Adobe® Acrobat® .pdf).

Data Evaluation

Field and laboratory data and findings will be compiled and reported to the City of Manchester and NHDES for analysis to determine whether additional remedial investigation or corrective action requirements are necessary. The data will be submitted per final report. The sediment data will also be included as part of the overall report for the project. Lab analyses data will be provided as Appendix C to this SAP.

The results of the parameters tested for will be compared to the sediment screening thresholds indicated in the table above following the steps outlined in NHDES Evaluation Of Sediment Quality Guidance Document (NHDES-WD-04-9) utilizing Component A of Triad Approach. These comparisons will dictate whether or not further action beyond this phase is required. NHDES will be consulted on the comparison with these thresholds.

The final report will include a summary of the activities completed including:

- actions that have been taken toward achieving project scope
- number and location of cores collected
- number of cores processed
- number of samples generated
- status of laboratory analysis
- all results of sampling, tests, and all other verified or validated data received or generated in the implementation of the work required
- identify any unresolved or uncompleted activities
- description of any outstanding issues and how they are being resolved

This project is a short duration project, therefore, no interim project status reports are required and will not be provided. The Data Summary Report prepared after the end of field activities will fully document the work and will include a summary of the work performed, a tabulation of results, field notes, processing data, chain-of-custody forms, and copies of the laboratory reports.

The project will be conducted upon final approval of this Sediment Sampling Plan by the NHDES and the associated Quality Assurance Project Plan by NHDES and the EPA.

4.0 SCHEDULE (To be completed after consultation with Lori Siegel and approval of plan)

Figure 3: Proposed sampling area at Station MP-S1 upstream of Maxwell Pond

Figure 4: Proposed sampling area at Station MP-S2 within the impoundment situated in the historical channel of Black Brook

Figure 5: Proposed sampling area at Station MP-S3 within the impoundment situated immediately upstream of the Maxwell Pond Dam

Figure 6: Proposed sampling area at Station MP-S4 downstream of the Maxwell Pond Dam and upstream of backwater influence from the Merrimack River

SEDIMENT SAMPLING PLAN (SSP)
APPENDICES

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APPENDIX A

SEDIMENT SAMPLING FIELD NOTES

FIELD LOG FORM

28 North Main Street
Randolph, Vermont 05060
Phone: (802) 728-3376
Fax: (802) 728-4930
www.dubois-king.com

SEDIMENT SAMPLING FIELD NOTES

Date of Observation:	
Weather:	
Town/City:	Manchester, NH
D&K Project No:	618517
Project Name:	Black Brook/Maxwell Pond Dam
D&K Representative(s):	
Time of Arrival:	
Time of Departure:	
D&K Project Manager:	Jeffrey Tucker, P.E.

OBSERVATIONS

Be sure to include: ***Detailed location of samples (mapped)***

Equipment used - include manufacturer, type, and serial number (if available)

Sampling Containers Used

Time each sample taken

Any necessary notes or supplemental forms used

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FIELD LOG FORM

28 North Main Street
Randolph, Vermont 05060
Phone: (802) 728-3376
Fax: (802) 728-4930
www.dubois-king.com

Date of Sample:	
Time of Sample:	
D&K Project No:	618517
Project Name:	Black Brook/Maxwell Pond Dam
Town/City:	Manchester, NH
D&K Project Manager:	Jeffrey Tucker, P.E.
D&K Sampler:	
Weather:	
Sample ID No.:	
Location (GPS coordinates (northing/easting) or Field Ties):	
Water Depth:	
Probing Depth:	
Sediment type (based on probing):	
Sediment description	
Sample type (composite, grab, etc)	
Approximate length of sediment core:	
Depth of penetration of the core recovered tube into the sediment:	

SEDIMENT SAMPLING PLAN (SSP)
APPENDIX B

ENDYNE 2007 QUALITY CONTROL DATA

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Endyne, Inc.
160 James Brown Drive
Williston Vermont

Control Limits 2006 EPA 8260B/624 - Ind. Standard as Percent Recovery - Solid Samples

Parameter	PRG mg/kg	Reporting Limit ug/Kg**	Method Detection Limit ug/Kg	MEAN Percent Recovery	Standard Deviation	Upper Warning Limit %	Lower Warning Limit %	Upper Control Limit %	Lower Control Limit %	Duplicate Difference 100ppb D/MS
2) Dichlorodifluoromethane	9.4E+04	50.0	3.37	92.7	14.5	121.8	63.7	136.3	49.1	30.2
3) Chloromethane	4.7E+04	30.0	4.64	96.8	13.7	124.2	69.3	138.0	55.5	30.4
4) Vinyl chloride	7.90E-02	20.0	5.04	99.6	14.6	128.8	70.5	143.3	55.9	33.7
5) Bromomethane	3.9E+03	50.0	12.5	102.4	12.3	126.9	77.8	139.2	65.6	33.2
6) Chloroethane	3.0E+03	50.0	5.62	103.0	13.8	130.5	75.5	144.2	61.7	31.5
7) Trichlorofluoromethane	3.9E+05	20.0	4.11	98.2	13.2	124.5	71.9	137.7	58.7	30.9
8) Diethyl ether	NA	50.0	4.70	103.3	14.0	131.4	75.3	145.4	61.3	29.3
9) 1,1-Dichloroethene	1.2E+05	10.0	2.83	101.3	13.1	127.4	75.2	140.4	62.1	32.5
10) Acetone	1.4E+07	100.0	19.8	105.6	14.0	133.5	77.7	147.5	63.7	48.0
11) Carbon disulfide	3.6E+05	50.0	6.36	100.1	14.1	128.3	71.9	142.4	57.8	31.3
12) Methylene chloride	9.1E+03	50.0	8.04	105.1	13.9	132.9	77.4	146.7	63.5	31.8
13) MTBE	3.2E+04	20.0	4.93	99.6	12.2	123.9	75.2	136.1	63.1	33.1
14) trans-1,2-Dichloroethane	NA	10.0	4.22	103.0	12.7	128.4	77.6	141.2	64.8	30.2
15) 1,1-Dichloroethane	5.1E+05	10.0	4.95	103.3	12.4	128.2	78.4	140.6	65.9	32.0
16) 2-Butanone	NA	100.0	7.39	105.3	14.8	134.9	75.7	149.7	60.9	55.4
17) 2,2-Dichloropropane	NA	10.0	3.32	98.0	12.3	122.6	73.4	134.9	61.1	29.3
18) cis-1,2-Dichloroethene	4.3E+04	10.0	3.85	100.6	10.7	122.0	79.3	132.6	68.6	23.8
19) Bromochloromethane	NA	20.0	2.95	101.0	11.9	124.7	77.3	136.6	65.4	24.7
20) Chloroform	2.2E+02	10.0	3.36	99.1	9.3	117.6	80.6	126.9	71.3	25.6
21) Tetrahydrofuran	9.4E+03	100.0	7.11	99.6	13.0	125.7	73.6	138.7	60.5	29.4
22) Dibromofluoromethane S1	surrogate	surrogate	surrogate	98.6	7.6	113.7	83.4	121.3	75.9	9.47
23) 1,1,1-Trichloroethane	1.2E+06	10.0	2.19	97.5	10.7	118.9	76.1	129.6	65.4	29.7
24) Carbon tetrachloride	2.5E+02	10.0	3.29	95.5	11.5	118.4	72.5	129.8	61.1	32.9
25) 1,1-Dichloropropene	NA	10.0	2.49	99.2	11.0	121.1	77.2	132.0	66.3	28.9
26) Benzene	6.4E+02	10.0	4.11	100.3	11.2	122.7	77.9	134.0	66.7	24.2

Endyne, Inc.
160 James Brown Drive
Williston Vermont

Control Limits 2006 EPA 8260B/624 - Ind. Standard as Percent Recovery - Solid Samples

Parameter	PRG mg/kg	Reporting Limit ug/Kg**	Method Detection Limit ug/Kg	MEAN Percent Recovery	Standard Deviation	Upper Warning Limit %	Lower Warning Limit %	Upper Control Limit %	Lower Control Limit %	Duplicate Difference 100ppb D/MS
27) 1,2-Dichloroethane	2.8E+02	10.0	3.78	98.1	9.3	116.7	79.4	126.1	70.1	23.6
29) Trichloroethene	5.3E+01	10.0	3.50	99.6	11.5	122.5	76.7	134.0	65.3	31.4
30) 1,2-Dichloropropane	3.4E+02	10.0	3.22	101.4	10.8	123.0	79.7	133.9	68.9	23.2
31) Dibromomethane	NA	20.0	3.90	97.4	8.8	115.0	79.9	123.7	71.2	22.8
32) Bromodichloromethane	8.2E+02	10.0	7.32	97.6	9.8	117.2	77.9	127.1	68.1	23.6
33) 2-Chloroethylvinylether	NA	200.0	3.33	97.7	15.8	129.4	66.0	145.2	50.2	40.3
34) cis-1,3-Dichloropropene	7.8E+02	10.0	5.30	98.5	8.9	116.3	80.7	125.2	71.8	22.0
35) MIBK	NA	100.0	3.29	97.4	13.6	124.6	70.2	138.2	56.6	27.4
36) Toluene-d8 S2	surrogate	surrogate	surrogate	99.0	3.3	105.6	92.5	108.8	89.2	2.36
37) Toluene	5.2E+05	10.0	4.57	100.6	9.8	120.2	81.1	129.9	71.3	26.2
38) trans-1,3-Dichloropropene	7.8E+02	10.0	4.61	96.7	9.5	115.6	77.7	125.1	68.2	22.7
39) 1,1,2-Trichloroethane	7.3E+02	10.0	4.14	97.8	9.5	116.9	78.7	126.4	69.2	23.2
40) Tetrachloroethene	4.8E+02	10.0	8.41	98.1	12.7	123.4	72.7	136.1	60.0	31.0
41) 1.3-Dichloropropane	1.0E+05	10.0	4.21	99.3	9.0	117.4	81.3	126.4	72.3	22.5
42) 2-Hexanone	NA	100.0	4.46	97.1	15.6	128.3	65.9	143.9	50.3	28.4
43) Dibromochloromethane	1.1E+03	10.0	3.40	94.5	9.8	114.1	75.0	123.8	65.2	22.1
44) 1,2-Dibromoethane	3.2E+01	20.0	2.95	97.5	8.7	114.9	80.1	123.6	71.4	23.6
46) Chlorobenzene	1.5E+05	10.0	2.96	100.8	8.0	116.7	84.9	124.6	76.9	19.2
47) Ethylbenzene	4.0E+05	10.0	5.43	101.1	9.6	120.3	82.0	129.8	72.4	23.2
48) 1,1,1,2-Tetrachloroethane	3.2E+03	10.0	3.15	98.8	8.3	115.3	82.2	123.6	74.0	19.1
49) m & p - Xylene	2.7E+05	20.0	2.67	101.4	9.6	120.6	82.2	130.2	72.6	48.6
50) o-Xylene	2.7E+05	20.0	2.54	100.7	8.5	117.8	83.6	126.4	75.1	22.0
51) Styrene	1.7E+06	10.0	1.87	101.6	7.9	117.4	85.8	125.3	77.9	20.6
52) Bromoform	6.2E+04	10.0	3.91	93.5	11.2	115.9	71.1	127.0	59.9	24.3
53) Isopropylbenzene	NA	10.0	3.32	100.8	10.6	122.0	79.5	132.6	68.9	27.3

Endyne, Inc.
160 James Brown Drive
Williston Vermont

Control Limits 2006 EPA 8260B/624 - Ind. Standard as Percent Recovery - Solid Samples

Parameter	PRG mg/kg	Reporting Limit ug/Kg**	Method Detection Limit ug/Kg	MEAN Percent Recovery	Standard Deviation	Upper Warning Limit %	Lower Warning Limit %	Upper Control Limit %	Lower Control Limit %	Duplicate Difference 100ppb D/MS
54) 4-Bromofluorobenzene S3	surrogate	surrogate	surrogate	98.5	5.5	109.4	87.5	114.9	82.0	3.73
55) 1,1,2,2-Tetrachloroethane	4.1E+02	20.0	4.89	100.5	11.4	123.4	77.6	134.9	66.2	23.4
56) Bromobenzene	2.8E+04	10.0	2.48	100.7	7.8	116.3	85.1	124.2	77.3	19.7
57) n-Propylbenzene	2.4E+05	10.0	2.78	101.1	10.8	122.7	79.4	133.6	68.6	27.9
58) 1,2,3-Trichloropropane	3.4E+01	20.0	2.46	100.5	10.5	121.6	79.4	132.1	68.9	23.1
59) 2-Chlorotoluene	NA	10.0	2.69	100.6	9.2	118.9	82.2	128.1	73.1	24.0
60) 1,3,5-Trimethylbenzene	2.1E+04	10.0	2.68	100.8	10.1	121.0	80.6	131.1	70.5	26.5
61) 4-Chlorotoluene	NA	10.0	2.08	100.3	9.2	118.7	81.8	128.0	72.5	23.6
62) tert-Butylbenzene	3.9E+05	10.0	2.91	100.6	11.5	123.5	77.7	135.0	66.2	29.4
63) 1,2,4-Trimethylbenzene	5.2E+04	10.0	3.49	100.4	9.2	118.8	82.0	128.0	72.8	24.9
64) sec-Butylbenzene	2.2E+05	10.0	2.48	102.0	11.8	125.6	78.4	137.5	66.5	30.3
65) 4-Isopropyltoluene	NA	10.0	3.65	100.9	11.9	124.7	77.0	136.6	65.1	29.9
66) 1,3-Dichlorobenzene	5.3E+05	10.0	3.39	101.3	8.6	118.5	84.1	127.1	75.5	22.3
68) 1,4-Dichlorobenzene	3.4E+03	10.0	6.54	101.5	8.3	118.1	84.8	126.4	76.5	21.2
69) n-Butylbenzene	2.4E+05	10.0	3.74	102.4	12.6	127.6	77.1	140.2	64.5	32.2
70) 1,2-Dichlorobenzene	6.0E+05	10.0	3.69	102.0	7.7	117.3	86.6	125.0	78.9	21.1
71) 1,2-Dibromo-3-chloropropane	4.6E+02	20.0	9.20	96.6	13.1	122.8	70.4	135.9	57.2	27.8
72) 1,2,4-Trichlorobenzene	6.2E+04	20.0	3.16	99.2	10.0	119.2	79.3	129.2	69.3	25.1
73) Hexachlorobutadiene	6.2E+03	20.0	0.510	99.4	14.8	129.0	69.8	143.7	55.1	35.4
74) Naphthalene	NA	20.0	0.867	100.2	13.0	126.1	74.2	139.0	61.3	31.0
75) 1,2,3-Trichlorobenzene	NA	20.0	0.694	100.4	10.3	121.0	79.8	131.3	69.5	25.4

** indicates values based on laboratory prepared samples (proper soil to methanol ratio) with a 100% solid value.

Endyne, Inc.
160 James Brown Drive
Williston Vermont

Control Limits 2006 EPA 8270C - Ind. Standard as Percent Recovery - Solid Samples								
Parameter	Method Detection Limit ug/L	Reporting Limit ug/L	MEAN	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
Pyridine	50.94	400.0	64.1	17.2	98.5	29.8	115.7	12.6
N-Nitrosodimethylamine	25.15	400.0	77.9	15.2	108.4	47.4	123.7	32.2
2-Fluorophenol (E.S.)		0.0	72.9	17.8	108.6	37.2	126.5	19.4
Aniline	21.84	400.0	79.9	18.5	116.8	43.0	135.3	24.5
Phenol	20.77	200.0	96.1	15.5	127.1	65.1	142.6	49.5
2-Chlorophenol	11.20	200.0	93.2	12.2	117.6	68.8	129.9	56.5
Phenol-d5 (E.S.)		0.0	86.9	17.9	122.6	51.2	140.5	33.3
bis(2-chloroethyl)Ether	22.75	200.0	89.5	16.3	122.1	56.9	138.5	40.5
1,3-Dichlorobenzene	11.92	80.0	76.6	18.5	113.7	39.6	132.2	21.0
1,4-Dichlorobenzene	7.88	80.0	75.3	16.8	108.9	41.7	125.6	25.0
1,2-Dichlorobenzene	14.44	80.0	77.6	16.9	111.5	43.7	128.4	26.8
Benzyl Alcohol	19.90	800.0	92.5	24.7	141.9	43.1	166.6	18.4
bis(2-chloroisopropyl	18.29	200.0	89.5	14.3	118.0	60.9	132.2	46.7
2-Methylphenol	13.66	200.0	101.4	12.2	125.8	76.9	138.0	64.7
Hexachloroethane	15.04	200.0	72.6	17.3	107.2	38.1	124.5	20.8
N-Nitrosodi-n-propylamine	20.78	400.0	100.3	18.0	136.4	64.3	154.5	46.2
3&4- Methylphenol	28.72	200.0	95.1	14.3	123.7	66.5	138.0	52.2
Nitrobenzene-d5 (E.S.)		0.0	84.1	21.8	127.8	40.4	149.6	18.5
Nitrobenzene	23.01	200.0	82.4	12.6	107.6	57.2	120.2	44.6
N-nitrosopiperidine	18.53	400.0	99.4	12.8	125.0	73.7	137.9	60.9
Isophorone	27.65	80.0	92.6	24.1	140.7	44.5	164.8	20.5
2-Nitrophenol	19.67	400.0	93.8	16.2	126.2	61.3	142.4	45.1
2,4-Dimethylphenol	31.27	200.0	97.3	16.4	130.1	64.5	146.5	48.1
bis(2-chloroethoxy)Methane	21.52	200.0	92.2	15.7	123.6	60.9	139.3	45.2
2,4-Dichlorophenol	18.44	200.0	92.8	19.1	130.9	54.7	149.9	35.6
1,2,4-Trichlorobenzene	7.15	80.0	79.6	17.2	113.9	45.2	131.0	28.1
Naphthalene	**9.77**	**80.0**	**85.9**	**8.9**	**103.6**	**68.2**	**112.5**	**59.4**
4-Chloroaniline	15.89	200.0	85.8	9.8	105.4	66.2	115.3	56.3
2,6-Dichlorophenol	18.99	200.0	99.4	13.1	125.5	73.2	138.6	60.1
Hexachlorobutadiene	13.41	200.0	82.0	19.9	121.7	42.3	141.6	22.5
N-Nitrosodi-n-butylamine	22.42	200.0	110.7	15.7	142.2	79.3	157.9	63.6
2-Methylnaphthalene	**15.37**	**80.0**	**89.7**	**20.0**	**129.7**	**49.7**	**149.8**	**29.6**
4-Chloro-3-methylphen	29.70	400.0	103.1	20.9	144.9	61.4	165.7	40.5
1-Methylnaphthalene	**16.74**	**80.0**	**98.5**	**14.4**	**127.3**	**69.7**	**141.7**	**55.4**
Hexachlorocyclopentad	16.72	800.0	75.1	17.9	111.0	39.2	128.9	21.2
2,4,6-Trichlorophenol	16.86	400.0	89.2	18.4	125.9	52.5	144.3	34.1
2,4,5-Trichlorophenol	28.47	400.0	85.6	18.1	121.7	49.4	139.8	31.4
2-Fluorbiphenyl (E.S.)		0.0	87.7	13.7	115.0	60.4	128.6	46.7

Endyne, Inc.
160 James Brown Drive
Williston Vermont

Control Limits 2006 EPA 8270C - Ind. Standard as Percent Recovery - Solid Samples								
Parameter	Method Detection Limit ug/L	Reporting Limit ug/L	MEAN	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
2-Chloronaphthalene	15.63	80.0	76.3	12.1	100.4	52.1	112.4	40.1
1-Chloronaphthalene	16.26	80.0	89.5	11.9	113.2	65.7	125.1	53.8
2-Nitroaniline	19.78	800.0	79.1	8.7	96.5	61.7	105.1	53.1
Acenaphthylene	**7.31**	**80.0**	**86.7**	**8.9**	**104.5**	**68.9**	**113.4**	**60.0**
Dimethylphthalate	12.58	200.0	96.2	14.6	125.3	67.1	139.9	52.5
2,6-Dinitrotoluene	18.83	200.0	88.4	17.5	123.5	53.3	141.0	35.7
Acenaphthene	**9.01**	**80.0**	**87.9**	**9.2**	**106.4**	**69.4**	**115.7**	**60.2**
3-Nitroaniline	17.01	800.0	88.4	14.2	116.9	60.0	131.1	45.7
2,4-Dinitrophenol	31.73	800.0	83.9	20.2	124.3	43.5	144.5	23.3
Dibenzofuran	9.93	80.0	83.9	13.3	110.5	57.3	123.8	44.0
2,4-Dinitrotoluene	26.33	200.0	100.9	25.3	151.5	50.2	176.9	24.8
1-Naphthylamine	5.34	400.0	89.8	24.0	137.7	41.9	161.6	17.9
2-Naphthylamine	12.21	400.0	86.8	24.3	135.4	38.3	159.7	14.0
Fluorene	**11.77**	**80.0**	**94.7**	**8.2**	**111.0**	**78.3**	**119.1**	**70.2**
4-Nitrophenol	36.04	400.0	93.0	24.3	141.5	44.4	165.7	20.2
Diethylphthalate	9.84	200.0	89.6	11.3	112.1	67.0	123.4	55.7
4-Chlorophenylphenylether	15.96	80.0	76.9	13.2	103.2	50.5	116.4	37.3
4-Nitroaniline	14.72	800.0	85.5	15.8	117.0	54.0	132.8	38.2
4,6-Dinitro-2-methylphenol	29.54	2000.0	101.8	22.5	146.8	56.8	169.3	34.3
Azobenzene	16.18	200.0	79.6	12.1	103.9	55.4	116.0	43.2
N-Nitrosodiphenylamine	8.34	200.0	90.8	10.3	111.4	70.3	121.7	60.0
2,4,6-Tribromophenol (E.S.)		0.0	91.4	18.3	128.1	54.7	146.4	36.4
4-Bromophenylphenylether	13.57	80.0	80.3	14.1	108.6	52.0	122.7	37.9
Hexachlorobenzene	12.71	200.0	78.8	14.3	107.5	50.2	121.8	35.9
Pentachlorophenol	20.00	800.0	76.0	17.2	110.4	41.6	127.7	24.3
Phenanthrene	**9.50**	**80.0**	**92.3**	**8.3**	**109.0**	**75.6**	**117.3**	**67.2**
Anthracene	**21.58**	**80.0**	**93.9**	**9.4**	**112.6**	**75.1**	**122.0**	**65.7**
Carbazole	64.83	400.0	91.0	13.8	118.5	63.4	132.3	49.6
Di-n-butylphthalate	27.42	200.0	104.7	11.0	126.8	82.6	137.8	71.6
Benzidine	6.54	400.0	41.4	10.7	62.9	20.0	73.6	9.3
Fluoranthene	**19.82**	**80.0**	**106.9**	**25.1**	**157.0**	**56.8**	**182.0**	**31.7**
Pyrene	**16.01**	**80.0**	**106.6**	**24.3**	**155.1**	**58.1**	**179.4**	**33.8**
Terphenyl-d14 (E.S.)		0.0	112.5	14.8	142.1	82.8	157.0	68.0
Butylbenzylphthalate	16.02	200.0	110.6	14.2	138.9	82.2	153.1	68.0
Benzo(a)anthracene	**13.51**	**80.0**	**97.7**	**9.9**	**117.4**	**78.0**	**127.3**	**68.1**
Chrysene	**18.37**	**80.0**	**100.9**	**9.4**	**119.6**	**82.1**	**129.0**	**72.7**
3,3'-Dichlorobenzidin	17.01	200.0	93.3	18.0	129.4	57.3	147.4	39.3
bis(2-ethylhexyl)phth	15.32	200.0	107.6	27.3	162.2	53.1	189.5	25.8

Endyne, Inc.
160 James Brown Drive
Williston Vermont

Control Limits 2006 EPA 8270C - Ind. Standard as Percent Recovery - Solid Samples								
Parameter	Method Detection Limit ug/L	Reporting Limit ug/L	MEAN	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
Di-n-octylphthalate	12.64	200.0	91.6	19.5	130.7	52.6	150.2	33.0
Benzo(b&k)fluoranthrenes	**11.96**	**80.0**	**93.5**	**8.7**	**110.9**	**76.1**	**119.6**	**67.4**
Benzo(a)pyrene	**19.82**	**80.0**	**92.3**	**8.6**	**109.5**	**75.1**	**118.1**	**66.5**
Indeno[1,2,3-cd]pyrene	**14.75**	**80.0**	**91.9**	**11.3**	**114.6**	**69.3**	**125.9**	**58.0**
Dibenz(a,h)anthracene	**17.37**	**80.0**	**91.0**	**11.8**	**114.5**	**67.4**	**126.2**	**55.7**
Benzo(g,h,i)perylene	**25.01**	**80.0**	**87.7**	**12.1**	**112.0**	**63.4**	**124.2**	**51.3**

Bolded parameters indicate PAH test group

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Endyne, Inc.
160 James Brown Drive
Williston, Vermont

Control Limits 2007 EPA 8081A - Ind. Standard as Percent Recovery - Solid Samples

Parameter	Method Detection Limit ug/kg	Reporting Limit ug/kg	MEAN ug/kg	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
BHC, alpha-	2.04	< 10.0	10.69	2.77	20.54	9.44	23.31	6.66
BHC, gamma-	1.76	< 10.0	11.05	2.75	20.98	9.97	23.73	7.21
BHC, beta-	1.88	< 10.0	12.50	3.17	23.61	10.92	26.78	7.75
BHC, delta-	2.55	< 10.0	12.86	2.63	21.96	11.44	24.59	8.81
Heptachlor	2.90	< 10.0	11.35	3.67	23.88	9.21	27.54	5.54
Aldrin	2.18	< 10.0	10.38	2.57	19.78	9.49	22.35	6.92
Hept Epoxide	2.42	< 10.0	16.58	2.71	22.00	11.15	24.72	8.44
Endosulfan I	2.64	< 10.0	16.65	2.57	21.80	11.50	24.37	8.93
DDE	2.26	< 10.0	17.07	2.67	22.41	11.72	25.09	9.05
Dieldrin	2.51	< 10.0	17.81	3.27	24.34	11.27	27.61	8.00
Endrin	2.40	< 10.0	17.71	3.06	23.82	11.59	26.87	8.54
Endosulfan II	2.78	< 10.0	17.16	3.42	24.00	10.31	27.42	6.89
DDD	5.41	< 10.0	17.90	3.07	24.05	11.76	27.12	8.69
Endrin Aldehyde	2.81	< 10.0	16.23	2.85	21.93	10.53	24.78	7.68
Endo Sulfate	4.27	< 10.0	17.03	3.63	24.30	9.77	27.93	6.13
DDT	3.51	< 10.0	18.08	3.27	24.63	11.54	27.90	8.26
Methoxychlor	5.39	< 20.0	18.61	2.97	24.55	12.66	27.52	9.69
Chlordane	2.34	< 20.0	66.60				86.6	46.6
Toxaphene	2.36	< 50.0	330				231	429
DCB (Surrogate)	NA	NA	30.60	4.09	38.783	22.417	42.874	18.326

Endyne, Inc.
160 James Brown Drive
Williston, Vermont

Control Limits 2007 EPA 8081A - Ind. Standard as Percent Recovery - Solid Samples

Parameter	Method Detection Limit ug/kg	Reporting Limit ug/kg	MEAN ug/kg	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
BHC, alpha-	2.04	< 10.0	10.69	2.77	20.54	9.44	23.31	6.66
BHC, gamma-	1.76	< 10.0	11.05	2.75	20.98	9.97	23.73	7.21
BHC, beta-	1.88	< 10.0	12.50	3.17	23.61	10.92	26.78	7.75
BHC, delta-	2.55	< 10.0	12.86	2.63	21.96	11.44	24.59	8.81
Heptachlor	2.90	< 10.0	11.35	3.67	23.88	9.21	27.54	5.54
Aldrin	2.18	< 10.0	10.38	2.57	19.78	9.49	22.35	6.92
Hept Epoxide	2.42	< 10.0	16.58	2.71	22.00	11.15	24.72	8.44
Endosulfan I	2.64	< 10.0	16.65	2.57	21.80	11.50	24.37	8.93
DDE	2.26	< 10.0	17.07	2.67	22.41	11.72	25.09	9.05
Dieldrin	2.51	< 10.0	17.81	3.27	24.34	11.27	27.61	8.00
Endrin	2.40	< 10.0	17.71	3.06	23.82	11.59	26.87	8.54
Endosulfan II	2.78	< 10.0	17.16	3.42	24.00	10.31	27.42	6.89
DDD	5.41	< 10.0	17.90	3.07	24.05	11.76	27.12	8.69
Endrin Aldehyde	2.81	< 10.0	16.23	2.85	21.93	10.53	24.78	7.68
Endo Sulfate	4.27	< 10.0	17.03	3.63	24.30	9.77	27.93	6.13
DDT	3.51	< 10.0	18.08	3.27	24.63	11.54	27.90	8.26
Methoxychlor	5.39	< 20.0	18.61	2.97	24.55	12.66	27.52	9.69
Chlordane	2.34	< 20.0	66.60				86.6	46.6
Toxaphene	2.36	< 50.0	330				231	429
DCB (Surrogate)	NA	NA	30.60	4.09	38.783	22.417	42.874	18.326

Endyne, Inc.
160 James Brown Drive
Williston, Vermont

Control Limits 2007 EPA 8081A - Ind. Standard as Percent Recovery - Solid Samples

Parameter	Method Detection Limit ug/kg	Reporting Limit ug/kg	MEAN ug/kg	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
BHC, alpha-	2.04	< 10.0	10.69	2.77	20.54	9.44	23.31	6.66
BHC, gamma-	1.76	< 10.0	11.05	2.75	20.98	9.97	23.73	7.21
BHC, beta-	1.88	< 10.0	12.50	3.17	23.61	10.92	26.78	7.75
BHC, delta-	2.55	< 10.0	12.86	2.63	21.96	11.44	24.59	8.81
Heptachlor	2.90	< 10.0	11.35	3.67	23.88	9.21	27.54	5.54
Aldrin	2.18	< 10.0	10.38	2.57	19.78	9.49	22.35	6.92
Hept Epoxide	2.42	< 10.0	16.58	2.71	22.00	11.15	24.72	8.44
Endosulfan I	2.64	< 10.0	16.65	2.57	21.80	11.50	24.37	8.93
DDE	2.26	< 10.0	17.07	2.67	22.41	11.72	25.09	9.05
Dieldrin	2.51	< 10.0	17.81	3.27	24.34	11.27	27.61	8.00
Endrin	2.40	< 10.0	17.71	3.06	23.82	11.59	26.87	8.54
Endosulfan II	2.78	< 10.0	17.16	3.42	24.00	10.31	27.42	6.89
DDD	5.41	< 10.0	17.90	3.07	24.05	11.76	27.12	8.69
Endrin Aldehyde	2.81	< 10.0	16.23	2.85	21.93	10.53	24.78	7.68
Endo Sulfate	4.27	< 10.0	17.03	3.63	24.30	9.77	27.93	6.13
DDT	3.51	< 10.0	18.08	3.27	24.63	11.54	27.90	8.26
Methoxychlor	5.39	< 20.0	18.61	2.97	24.55	12.66	27.52	9.69
Chlordane	2.34	< 20.0	66.60				86.6	46.6
Toxaphene	2.36	< 50.0	330				231	429
DCB (Surrogate)	NA	NA	30.60	4.09	38.783	22.417	42.874	18.326

Endyne, Inc.
160 James Brown Drive
Williston, Vermont

Control Limits 2007 EPA 8082 - Ind. Standard as Percent Recovery - Aqueous Samples

Parameter	Method Detection Limit ug/L	Reporting Limit ug/L	MEAN	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
Arochlor-1016	0.152	< 0.50	2.94	0.56	4.06	1.82	4.63	1.26
Arochlor-1221		< 0.50	3.00				3.60	2.40
Arochlor-1232		< 0.50	3.00				3.60	2.40
Arochlor-1242		< 0.50	3.00				3.60	2.40
Arochlor-1248		< 0.50	3.00				3.60	2.40
Arochlor-1254		< 0.50	3.00				3.60	2.40
Arochlor-1260	0.111	< 0.50	3.03	0.41	3.99	2.06	4.47	1.58
Arochlor-1268		< 0.50	3.00				3.60	2.40
DCB (Surrogate)	NA	NA	0.950	0.170	1.286	0.604	1.456	0.434

Control Limits 2007 EPA 8082 - Ind. Standard as Percent Recovery - Solid Samples

Parameter	Method Detection Limit ug/kg	Reporting Limit ug/kg	MEAN ug/kg	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
Arochlor-1016	3.37	< 50.0	2.89	0.55	3.99	1.78	4.54	1.23
Arochlor-1221		< 50.0	100				120	80
Arochlor-1232		< 50.0	100				120	80
Arochlor-1242		< 50.0	100				120	80
Arochlor-1248		< 50.0	100				120	80
Arochlor-1254		< 50.0	100				120	80
Arochlor-1260	1.96	< 50.0	2.82	0.36	3.54	2.09	3.91	1.72
Arochlor-1268		< 50.0	100				120	80
DCB (Surrogate)	NA	NA	0.93	0.27	1.469	0.381	1.741	0.109

Endyne, Inc.
160 James Brown Drive
Williston, Vermont

2006 Quality Control for PRIORITY POLLUTANT 13 METALS in Water

Element	Method	MDL ug/L	Reporting Limit ppb	Independent LFB Accuracy Limit %	Precision Limit %
Antimony	SM 3113B	0.82	2	10	20
Arsenic	SM 3113B	0.57	2	10	20
Beryllium	EPA 6010	0.3	2	10	20
Cadmium	EPA 6010	0.4	3	10	20
Chromium	EPA 6010	4.4	10	10	20
Copper	EPA 6010	2.43	10	10	20
Lead	SM 3113B	0.26	2	10	20
Mercury	EPA 7470	0.22	1	10	20
Nickel	EPA 6010	5.17	20	10	20
Selenium	SM 3113B	1.73	5	10	20
Silver	EPA 6010	4.53	10	10	20
Thallium	SM 3113B	0.36	1	10	20
Zinc	EPA 6010	2.16	20	10	20

2006 Quality Control for PRIORITY POLLUTANT 13 METALS in Soil

Element	Method	MDL mg/Kg	Reporting Limit mg/kg	Independent LFB Accuracy Limit %	Duplicate Precision Limit %
Antimony	EPA 6010	1.09	2.00	10.00	20.00
Arsenic	EPA 6010	0.676	2.00	10.00	20.00
Beryllium	EPA 6010	0.144	0.50	10.00	20.00
Cadmium	EPA 6010	0.150	0.50	10.00	20.00
Chromium	EPA 6010	0.257	1.00	10.00	20.00
Copper	EPA 6010	0.235	1.00	10.00	20.00
Lead	EPA 6010	0.468	2.00	10.00	20.00
Mercury	EPA 7471	0.300	0.600	10.00	20.00
Nickel	EPA 6010	0.334	1.50	10.00	20.00
Selenium	EPA 6010	1.01	3.00	10.00	20.00
Silver	EPA 6010	0.373	2.00	10.00	20.00
Thallium	EPA 6010	0.938	1.00	10.00	20.00
Zinc	EPA 6010	0.373	1.50	10.00	20.00

SEDIMENT SAMPLING PLAN (SSP)
APPENDIX C

ENDYNE ANALYSIS DATA
(ONCE COMPLETE)

APPENDIX B

MANCHESTER URBAN POND RESTORATION PROGRAM YEAR 2 REPORT – MAY 2002 (2001 SEDIMENT DATA)

From: Manchester Urban Ponds Report 2001

Maxwell Pond

Dissolved Oxygen

Dissolved oxygen levels were relatively high in relation to other Manchester ponds due to the stream-like characteristics of Maxwell Pond. The lowest dissolved oxygen saturation recorded at Maxwell Pond was 43.0% at the pond's deepest point. DO levels in 2001 were very similar to those found in 2000.

Total Phosphorus

Due to the fact that the deepest spot in Maxwell Pond is 1.1 meters, there was no thermal stratification, so only "surface grab" samples were necessary for in-pond sampling. Total phosphorus concentrations ranged from 0.013 to 0.023 mg/L, with an average of 0.018 mg/L. According to data from DES Trophic Classification of NH Lakes and Ponds, 1981, Maxwell Pond TP concentration was 0.018 mg/L. These two years of data (1981 and 2001) are similar but higher than 2000 levels. This shows fluctuations in TP concentrations over the years. Due to the high turnover of pond volume and shallowness here, inlet samples are especially important. TP concentrations in the inlet samples (Black Brook) never rose above 0.016 mg/L and averaged 0.012 mg/L. This is slightly lower than inlet TP concentrations in 2000.

Conductivity

Conductivity of Maxwell Pond ranged from 124.1 to 206.0 uMhos/cm, with an average of 154.6 uMhos/cm. DES 1981 data shows conductivity at 56.0 uMhos/cm. 2001 saw a 22% rise in conductivity over 2000, and a 176% rise over 1981. Inlet samples ranged from 122.4 to 233.0 uMhos/cm and averaged 167.6 uMhos/cm in 2001. This is a 28% rise from 2000. Clearly, something is causing a significant increase in conductivity at Maxwell Pond. Future investigations will attempt to identify the cause(s) of the increase in conductivity.

Chlorophyll-*a*

In-pond chlorophyll *a* concentrations were very low, ranging from 1.11 to 5.45 mg/m^3, and averaging 3.17 mg/m^3. These low readings are most likely due to the pond's high flushing rate. Though still relatively low, the 2001 average is approximately double the 2000 average.

Transparency

As the bottom could clearly be seen at 1.1 meters, Secchi disk transparency was >1.1 meters and could not be measured more accurately due to lack of depth.

pH and Acid Neutralizing Capacity

The pH of Maxwell Pond ranged from 6.45 to 6.87, averaging 6.63. 2000 pH readings at Maxwell Pond were similar to those found in 2001. This is slightly low for NH freshwater ecosystems, but still well within the range for supporting aquatic life. pH readings by NH DES in 1981 were similar at 6.4. ANC was also consistently lower than other Manchester ponds, ranging from 4.0 to 16.7 mg of CaCO/L, with an average of 9.8 mg/L. In 1981, NH DES found ANC to be 6.4 mg/L. Maxwell is therefore less able to buffer acidic inputs, which may help explain the low pH readings.

Turbidity

Turbidity in Maxwell Pond ranged from 0.94 to 4.3 (NTU) and averaged 3.39 (NTU). This is a slight increase from 2000 when the average turbidity was 2.03 (NTU). NH DES 1981 turbidity readings were a bit higher at 4.3 (NTU).

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Maxwell Pond appears to be showing a slight trend of decreasing water quality since 2000. Two years of data, however, do not accurately represent a trend. Natural fluctuations, upstream disturbances and discharges, and precipitation variations could all be singled out as reasons for water quality fluctuations.

Overall Water Quality

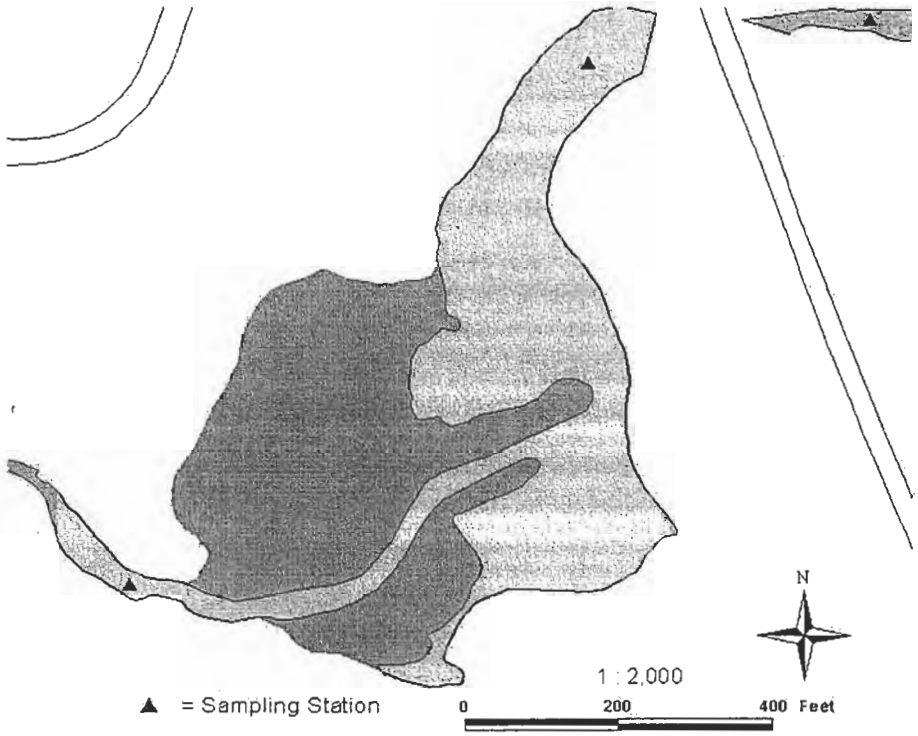
The water quality of Maxwell Pond is better than any other Manchester Pond. Maxwell has a very high turnover rate and relatively little urban development in the watershed. Its streamlike characteristics allow most pollutants to wash downstream. Rapid sedimentation, due to the dam, and vegetation growth is occurring in some parts of the pond, however.

Table 5[1]
Comparison of Maxwell Pond – 1981*, 2000 & 2001

		2000		2001	
Parameter	1981	Mean	Median	Mean	Median
pH	6.4	6.54	6.55	6.63	6.62
Alkalinity (mg/L)	7.0	6.8	6.9	9.8	9.6
Total phosphorus (mg/L)	.018	.014	.014	.018	.018
Conductivity (uMhos/cm)	56.0	121.8	127.3	154.6	148.5
Secchi disk (m)	>1.2	>1.1		>1.1	

1) All values are epilimnetic.
* NH Dept. of Environmental Services. 1981. Trophic Classification of NH Lakes and Ponds.

Figure 3 – Maxwell Pond Sampling Stations



Vegetation Inventory

A vegetation inventory was conducted at Maxwell Pond on August 3 and August 27, 2001. The following is a list of exotic plant species found at Maxwell Pond. Exotic species represent a threat to the ecological integrity of a pond ecosystem. A complete native plant inventory list is included in Appendix C.

Emergent

- Purple Loosestrife *(Lythrum salicaria)**

Upland Shrub

- European Buckthorn *(Rhamnus frangula)**
- Honeysuckle *(Lonicera spp.)**
- Japanese or European Barberry *(Berberis vulgaris)**
- Oriental Bittersweet *(Celastrus orbiculata)**

Maxwell Pond riparian areas are dominated by invasive wetland and upland plant species.

Sediment Sample Analysis

Maxwell Pond sediment core samples showed elevated levels of chromiun, copper, lead, manganese, mercury, nickel and zinc. All of these metals exceeded the "lowest effects level" but not the "severe effects level" criteria for aquatic organisms. The pesticide DDT constituent, p,p'-DDE also exceed the "lowest effects level" for aquatic organisms.

Current Status

Plans are currently being discussed for possible dam removal at Maxwell Pond. This would drastically alter Maxwell Pond as we know it. In partnership with DES and Trout Unlimited the UPRP will assist with a feasibility study at Maxwell Pond to determine baseline conditions, and formulate hypotheses regarding the reaction of Black Brook to dam removal. Identification of existing channel location and conditions as well as historic, pre-dam channel characteristics is crucial to understanding the long term effects that dam removal may have on this site and the Black Brook corridor as a system. The dam removal study workplan will include aerial topographic surveying, stream channel morphology study, bathymetric survey and sediment depth mapping of Maxwell Pond, water quality monitoring of Maxwell Pond, and biomonitoring of Black Brook including macroinvertebrate surveys and fish surveys. If the dam is removed, approximately six miles of free-flowing stream would be restored.

Trout Unlimited was awarded a $13,850 grant from the NH DES local watershed initiative grant program to conduct the first phase of the Black Brook corridor study: photogrammetric mapping. The product will be an up-to-date aerial topographic map accurate to a contour interval of one foot.

Concurrent with dam removal study, a restoration plan is being created for a disturbed site upstream of Maxwell Pond. A concrete aggregate and transportation operation has been impacting Black Brook for several years. Impacts include channel obstruction and filling as well as sedimentation and artificial bank armoring. The property owner has been cooperating with DES authorities to remedy the problems on the site, as well as reconfigure stream crossings to allow proper fish passage and possibly relocate the stream channel to its historic location.

Maxwell Pond – native plant species

Inventoried on 8/3/2001 by Jen Drociak of the Manchester Conservation Commission.

Emergent

Arrowhead *(Sagitaria latifolia)*
Burreed *(Sparganium eurycarpum)*
Pickerelweed *(Pontederia cordata)*
Pipewort *(Eriocaulon septangulare)*

Floating

Floating Heart
Floating Pondweed *(Pontamogeton natans)*
Ribbon-Leaf Pondweed *(Pontamogeton epihydrus)*
Yellow Pond Lily *(Nuphar spp.)*

Submerged

Bladderwort *(Utricularia vulgaris)*
Bushy Pondweed/Naiad *(Najas flexilis)*

Wetland Herb

Blue Flag/Iris *(Iris versicolor)*
Cinnamon Fern *(Osmunda cinnamomea)*
Marsh St. Johnswort *(Hypericum virginicum)*
Meadowsweet *(Spiraea latifolia)*
Sedge *(Carex spp.)*
Sensitive Fern *(Onoclea sensibilis)*
Soft-Stemmed Bulrush *(Scirpus validus)*
Steeplebush *(Spiraea tomentosa)*
Three-Way Sedge *(Dulichium arundinaceum)*
Water Smartweed *(Polygonum amphibium)*
Water Hemlock *(Cicuta malculata)*

Wetland Shrub

Buttonbush *(Cephalanthus occidentalis)*
Northern Arrowwood *(Viburnum recognitum)*
Speckled Alder *(Alnus rugosa)*

Upland Shrub

Red-Osier Dogwood *(Cornus stolinifera)*

Upland Herb

Canada Mayflower (Mainthemum canadense)
Climbing Hempweed *(Mikania scandens)*
False Solomon's Seal *(Smilacina racemosa)*
Hay-Scented Fern *(Dennstaedtia punctilobulia)*
Milkweed *(Asclepias syriaca)*

McQuesten Pond – native plant species

Inventoried on 8/3/2001 by Jen Drociak of the Manchester Conservation Commission.

Emergent

Arrowhead/Duck-potato *(Sagitaria latifolia)*
Cattail *(Typha latifolia)*

Floating

Duckweed *(Lemna spp.)*

Wetland Shrub

Speckled Alder *(Alnus rugosa)*

Wetland Herb

Blue Vervain *(Verbena hastate)*
Jewelweed *(Impatiens capensis)*
Meadowsweet *(Spiraea latifolia)*

Wetland Tree

Red Maple *(Acer rubrum)*
Willow *(Salix spp.)*

Upland Herb

Common Mullein *(Verbascum thaspus)*
Nightshade *(Atropa belladonna)*
Queen Anne's Lace *(Daucus carota)*
Yarrow *(Achillea millefolium)*

Upland Tree

Ashed-Leaved Maple/Boxelder *(Acer negunda)*
Black Cherry *(Prunus serotina)*
Quaking Aspen *(Populus tremuloides)*
Sassafras *(Sassafras albidum)*
White Birch *(Betula papyrifera)*

Sediment Metals Screening Summary Table
Maxwell Pond

All Values Listed are in micrograms per kilogram (ppm)

Metals	Maxwell Pond	OSWER Type	LEL	SEL
Aluminum (Al)	54,500			
Antimony (Sb)	<5			
Arsenic (As), Total	<3	8.2	6	33
Barium (Ba)	377			
Beryllium (Be)	3.7			
Calcium (Ca)	8,200			
Cadmium (Cd)	<1	1.2	0.6	10
Chromium (Cr), Total	**31**	81	26	110
Cobalt (Co)	20			
Copper (Cu)	**29.8**	34	16	110
Iron (Fe)	2.1		20,000	40,000
Lead (Pb)	**108**	47	31	250
Manganese (Mn)	**965**		460	1100
Magnesium (Mg)	3,700			
Mercury (Hg), Total	**0.81**	0.15	0.2	2
Nickel (Ni)	**21**	21	16	75
Silver (Ag)	0.3			
Vanadium (V)	46			
Zinc (Zn)	**144**	150	120	820

LEGEND

Sediment Criteria

OSWER	U.S. EPA (1996). Ecotox Thesholds
LEL	Lowest Effects Level (Jaagumagi et al. 1995)
SEL	Severe Effects Level (Jaagumagi et al. 1995)

Pesticide PCB Calculation Sheet

Site Name	Maxwell Pond			
Site Description		N 43 00' 37.02"	W 71 28' 46.01"	
Site Number	M1			
TOC Adjusted ECOTOX Threshold		LEL	SEL*	SQAL/SQC*
Aldrin	ND	0.002	0.208	
BHC, alpha-	ND	0.006	0.26	
BHC, beta-	ND	0.005	0.546	
BHC, delta-	ND			0.338
BHC, gamma- (Lindane)	ND	0.003	0.026	0.00962
Chlordane, alpha(cis)-	ND			
Chlordane, gamma(trans)-	ND			
Chlordane, technical grade	ND	0.007	0.156	
DDD, p, p'-	ND	0.008	0.156	
DDE, p,p'-	**0.011**	0.005	0.494	
DDT (Total)	ND	0.007	0.312	
Dieldrin	ND	0.002	2.366	0.286
Endosulfan I (alpha)	ND			0.00754
Endosulfan II (beta)	ND			0.0364
Endosulfan sulfate (sulfite?)	ND			
Endrin	ND	0.003	3.38	0.1092
Endrin aldehyde	ND			
Endrin ketone	ND			
Heptachlor	ND			
Heptachlor epoxide	ND	0.005	0.13	
Methoxychlor	ND			0.0494
Toxaphene	ND			0.1
PCB (Aroclor-1016)	ND	0.007	1.378	
PCB (Aroclor-1221)	ND			
PCB (Aroclor-1232)	ND			
PCB (Aroclor-1242)	ND			
PCB (Aroclor-1248)	ND	0.03	3.9	
PCB (Aroclor-1254)	0.036J, B	0.06	0.884	
PCB (Aroclor-1260)	ND	0.005	0.624	
LEGEND	J = Below Detection Limit		B = Present in Blank	

TOC Adjusted Sediment Criteria

LEL	Lowest Effects Level (Jaagumagi et al. 1995)
SEL*	Severe Effects Level (Jaagumagi et al et al. 1995)
SQAL/SQC*	

Sediment Quality Advisory Levels/Sediment Quality Criteria
(values are lower limit of 95% confidence limit) (USEPA, 1997)

SEL and SQAL/SQC adjusted for TOC
ND=Not detected
Sediment Values in (ppm) or *ug/g organic carbon for SEL and SQAL/SQC

PAH SEL Calculation Sheet

Site Name	Maxwell Pond			
Site Description	N 43 00' 37.02" W 71 28' 46.01"			
Site Number	M1			
TOC Adjusted ECOTOX Threshold		LEL	SEL*	SQAL/SQC*
Methylnaphthalene, 2-	ND			
Acenaphthene	ND			3.38
Acenaphthylene	ND			
Anthracene	ND	0.22	9.62	
Benzo(a)anthracene	ND	0.32	38.48	
Benzo(b)fluoranthene	0.2J			
Benzo(k)fluoranthene	ND	0.24	34.84	
Benzo(a)pyrene	0.17J	0.37	37.44	
Benzo(ghi)perylene	ND	0.32	8.32	
Chrysene	0.15J	0.34	11.96	
Dibenzo(a,h)anthracene	ND	0.06	3.38	
Fluoranthene	0.27J	0.75	26.52	16.12
Fluorene	ND	0.19	4.16	1.404
Indeno(1,2,3-cd)pyrene	ND	0.2	8.32	
Naphthalene	ND			1.222
Phenanthrene	0.130J	0.56	24.7	1.8
Pyrene	0.25J	0.49	22.1	
LEGEND	J = Below Detection Limit			

TOC Adjusted Sediment Criteria

LEL	Lowest Effects Level (Jaagumagi et al. 1995)
SEL*	Severe Effects Level (Jaagumagi et al et al. 1995)
SQAL/SQC*	Sediment Quality Advisory Levels/Sediment Quality Criteria (values are lower limit of 95% confidence limit) (USEPA, 1997)

SEL and SQAL/SQC adjusted for TOC
ND=Not detected
Sediment Values in (ppm) or *ug/g organic carbon for SEL and SQAL/SQC

SEDIMENT CORE METALS ANALYSIS

Sample Ident	Be	Na	Mg	Al	P	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Sr
Scheme Code	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80
Analysis Unit	ppm	%	%	%	%	%	%	ppm	%	ppm	ppm	ppm	%	ppm	ppm	ppm	ppm	ppm	ppm
Detection Limit	0.5	0.01	0.01	0.01	0.01	0.01	0.01	0.5	0.01	2	1	2	0.01	1	1	0.5	0.5	3	0.5
Dorrs Pond 1(deep)	5	13000	4700	62400	2300	18800	7400	7.4	3200	92	54	765	4.73	31	60	78.1	786	18	118
Dorrs Pond 2(north)	5.9	12100	4300	56100	3500	17800	8400	6.4	2800	66	75	**3090**	9.37	47	**75**	**130**	**2070**	25	124
Maxwell Pond	3.7	13800	3700	54500	900	17300	8200	5.9	3300	46	31	965	2.1	20	21	29.8	144	<3	148
Nutts Pond 1 (south)	3.4	13100	6000	55200	1900	18800	7800	7.3	3100	94	**122**	807	5.62	18	37	**232**	**1220**	**94**	119
Nutts Pond 2(deep)	3.3	14000	6300	56100	4400	17000	6400	8	2800	129	**110**	1090	6.4	32	59	**214**	**1590**	**38**	100
Nutts Pond 3(north)	3.5	15600	5700	58600	1300	21800	9100	6.4	3300	107	63	531	2.34	11	37	**159**	**1000**	6	146
Pine Island Pond 1(deep)	3.8	15700	4700	63100	1200	22100	9300	7.2	3800	63	41	587	2.34	9	17	58.9	147	<3	159
Pine Island Pond 2(north)	2.6	15500	2400	47600	600	16300	9500	4.7	3200	27	20	753	1.25	5	10	12.2	68.1	<3	149
Stevens Pond 1(deep)	3.2	11900	4500	48100	2200	14700	7900	5.8	2400	79	50	866	5.23	17	34	76.7	**927**	4	104
Stevens Pond 2(north)	2.5	11600	4200	42700	1100	13500	15700	5.3	2300	50	103	978	3.81	10	48	**157**	431	<3	152
Duplicate-1	5.5	14100	4900	68100	2500	2.02	8100	7.9	3500	99	51	831	5.1	31	65	80.9	761	16	130

Y	Zr	Mo	Ag	Cd	Sn	Sb	Ba	La	W	Pb	Bi	Li	Hg
ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	ICP80	CHM-20
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppb
0.5	0.5	1	0.2	1	10	5	1	0.5	10	2	5	1	5
18.7	29.8	4	0.7	3	11	<5	405	39.2	<10	**363**	<5	59	**213**
35.2	64.3	4	1	6	<10	<5	420	58.9	<10	**294**	<5	48	155
11.6	28.8	2	0.3	<1	<10	<5	377	30.6	<10	108	<5	33	81
20.5	74.9	8	1.1	5	11	<5	394	36.6	21	**387**	<5	45	**208**
33.7	49.9	14	0.6	9	14	7	442	35.1	72	**577**	<5	49	**503**
49.1	66.1	8	<0.2	3	<10	8	440	34.8	17	**311**	<5	42	**331**
13.4	48.4	3	0.3	<1	<10	<5	478	38.8	<10	76	<5	35	79
9.7	26.6	2	<0.2	<1	<10	<5	350	20.4	<10	36	<5	15	24
13.9	18.2	4	0.2	3	<10	<5	362	34.6	<10	213	<5	43	166
14.6	34.4	5	0.6	1	<10	<5	348	33.5	<10	129	<5	27	92
20.5	34.3	4	0.8	3	13	<5	435	45.1	<10	**396**	<5	63	**226**

APPENDIX C

NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM (NELAP)

Friday, Mar. 16, 2007

New Hampshire Department of Environmental Services

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Environmental Laboratory Accreditation Program

The New Hampshire Environmental Laboratory Accreditation Program provides accreditation to environmental laboratories in New Hampshire, as well as out of state, to ensure sufficiently accurate, precise, and consistent results of tests, analyses, and measurements.

New Hampshire Environmental Laboratory Accreditation Program, NH ELAP, is currently accrediting environmental laboratories according to the July 2003 edition of the National Environmental Laboratory Accreditation Conference (NELAC) Standards.

NH ELAP conducts On-Site Assessments of the laboratories, which are granted primary accreditation. Accreditation is based upon the results of the assessment and the results of the laboratory's participation in proficiency test studies. Laboratories granted secondary accreditation by NH ELAP are assessed by other NELAP recognized accrediting authorities using the NELAC Standards. Secondary Accreditation is based upon the accreditation granted by the Primary Accrediting Authority.

NH ELAP...

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NELAP RECOGNIZED

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Friday, Mar. 16, 2007

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Laboratory Services

Laboratory Overview

The DES laboratory is a National Environmental Laboratory Accreditation Program (NELAP) accredited laboratory. Twice each year the lab participates in a series of performance evaluations to test its capability to accurately determine many analytical parameters. Based on the accuracy of these performance tests, along with on-site audits of the laboratory processes, the DES laboratory has maintained full accreditation for drinking water and wastewater parameters. Please see the **full list of all parameters** for which the DES laboratory is currently accredited.

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For Public Water Supplies
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Test Containers

What We Do

The DES laboratory is a full service laboratory offering testing for microbiology, organic chemistry, inorganic chemistry and radiochemistry. The microbiology section checks for bacterial organisms that can be found primarily in water. Total Coliform and *E. Coli* are the primary bacterial indicators. The organic chemistry section conducts analyses on chemicals containing the element carbon. Volatile Organic Compounds (VOCs) - including the gasoline constituent methyl tertiary butyl ether (MtBE), - other petroleum products, industrial solvents, pesticides and herbicides are common tests. The inorganic chemistry section analyzes for the non-carbon elements and associated compounds found in our environment. These tests include metals such as Arsenic and Lead, nutrient compounds containing Nitrogen and Phosphorus, and physical measurements such as pH and specific conductance. The radiochemistry section conducts analyses of those inorganic elements, primarily Uranium and Radon, that are radioactive.

The DES laboratory is the primacy lab in New Hampshire for the U.S. Environmental Protection Agency (EPA). This designation requires the laboratory to maintain the capability and capacity to carry out specific methods for all the Safe Drinking Water Act parameters for enforcement cases and emergency situations.

Homeowner Water Testing

The U.S. Environmental Protection Agency and the Department of Environmental Services encourage all New Hampshire homeowners who have their own well to test their water periodically. Our jointly

published guide is available on line. Testing for all the recommended parameters is available at the DES laboratory on a fee for service basis. Upon completion of each analysis homeowners receive a full report along with an interpretation of test results. In addition, staff are available to provide an unbiased assessment of the water quality and to provide further information on available references and treatment options. Other environmentally specific situations and related laboratory services should be discussed with the laboratory directly. For more information **e-mail** or call us at 271-3445.

View PDF files with Adobe Acrobat Reader, available for free download at **Adobe.com**

Updated: February 2006

NH.Gov | Privacy Policy | Accessibility Policy

APPENDIX D

SEDIMENT SAMPLING FIELD NOTES

FIELD LOG FORM

28 North Main Street
Randolph, Vermont 05060
Phone: (802) 728-3376
Fax: (802) 728-4930
www.dubois-king.com

SEDIMENT SAMPLING FIELD NOTES

Date of Observation:	
Weather:	
Town/City:	Manchester, NH
D&K Project No:	618517
Project Name:	Black Brook/Maxwell Pond Dam
D&K Representative(s):	
Time of Arrival:	
Time of Departure:	
D&K Project Manager:	Jeffrey Tucker, P.E.

OBSERVATIONS

Be sure to include: ***Detailed location of samples (mapped)***

Equipment used - include manufacturer, type, and serial number (if available)

Sampling Containers Used

Time each sample taken

Any necessary notes or supplemental forms used

PDF processed with CutePDF evaluation edition www.CutePDF.com

FIELD LOG FORM

28 North Main Street
Randolph, Vermont 05060
Phone: (802) 728-3376
Fax: (802) 728-4930
www.dubois-king.com

Date of Sample:	
Time of Sample:	
D&K Project No:	618517
Project Name:	Black Brook/Maxwell Pond Dam
Town/City:	Manchester, NH
D&K Project Manager:	Jeffrey Tucker, P.E.
D&K Sampler:	
Weather:	
Sample ID No.:	
GPS coordinates (northing/easting):	
Water Depth:	
Probing Depth:	
Sediment type (based on probing):	
Sediment description	
Sample type (composite, grab, etc)	
Approximate length of sediment core:	
Depth of penetration of the core recovered tube into the sediment:	

APPENDIX E
ENDYNE, INC. QUALITY SYSTEMS MANUAL

Endyne, Inc.

160 James Brown Dr.
Williston, VT 05495
Phone: (802) 879-4333
Fax: (802) 879-7103

QUALITY SYSTEMS MANUAL

REVISED: September 20, 2005
Revision #: 7

Technical Director: _____Date : _____
Harry B. Locker Ph.D.

QA Officer: _____Date : _____
Mark L. Fausel

Document Control Copy on Buff colored paper. Any other color is not official version

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1.0 Quality Policy Statement

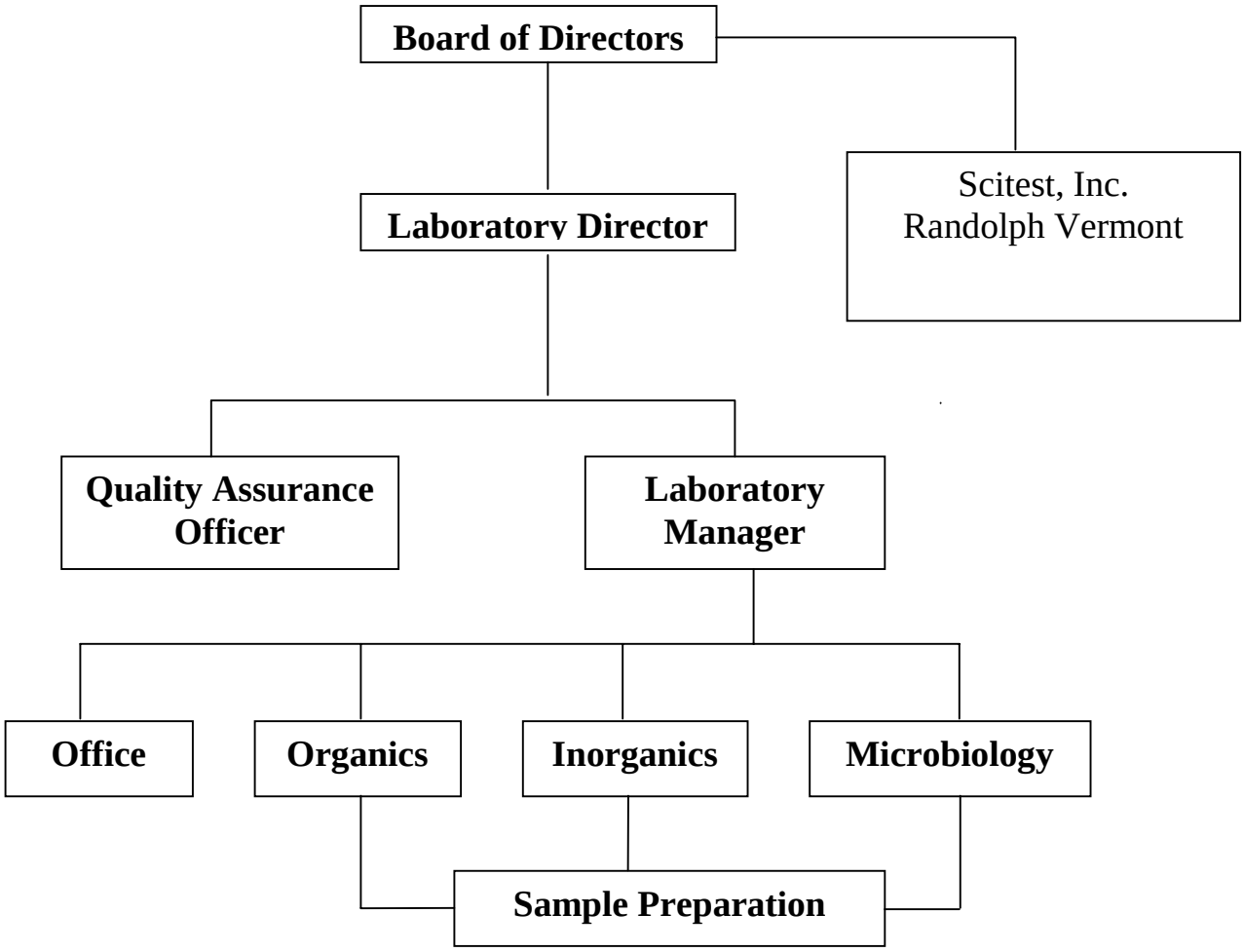
Endyne, Inc. is committed to providing consistent, high quality data in a timely manner. It is our belief that each individual analytical result generated by Endyne is of critical importance to our client. It is thus imperative that Endyne be able to generate and report data of known quality.
To that end, we have outlined our thorough Quality Systems Protocols with the intent of clarifying for our clients all the procedures and precautions that insure the highest level of quality data. Demonstration of Endyne's commitment to maintain our quality objective is evidenced in the following.

- An expertly staffed and fully equipped Laboratory facility.
- Successful participation in multiple EPA/NELAC approved proficiency testing programs.
- Successful implementation of a NELAC compliant quality system.
- Annual internal audits with management review.
- Timely reporting of analytical results.
- Laboratory test results that are supported by quality control data and documented testing procedures.

This quality policy is communicated to all new employees. It is understood, implemented, and maintained by employees at all levels. This is documented by management through the employee evaluation process, the training procedure, the internal audit process, and the document control process.

2.0 Organization Structure/Personnel

The laboratory is comprised of 3 major analytical divisions; Organic, Inorganic and Microbiological. Analysts in each division are trained to perform a variety of analyses and each analysis has at least two analysts trained and qualified to perform the procedure. Laboratory management is comprised of three positions; Laboratory Director, Laboratory Manager and QA Officer. There are two auxiliary divisions which assist in the overall sample flow within the laboratory; Sample Preparation and Office Staff.



2.1 Position Responsibilities

2.1.1 Organic and Inorganic Analysts are responsible for maintaining their own method workloads. Analysts record data and individual QA/QC results in the appropriate databases. Analysts trained in a method perform the initial data quality review of other analysts' work. The QA Officer who reviews the resultant data with the Laboratory Director compiles this information. Analysts report to the Laboratory Manager.

2.1.2 The Chief Microbiologist performs all microbiology and related abilities. The Chief Microbiologist receives training and education necessary to meet certification requirements. The Chief Microbiologist reports directly to the Laboratory Manager.

2.1.3 The Sample preparation department is responsible for all sample preservation and storage at the laboratory. This includes dispersing sample aliquots to the different divisions of the laboratory and ensuring there is sufficient sample volume to perform all requested analyses. Sample technician prepares all sample containers for shipment, including preservation and sampling instructions. The sample tech is also responsible for assigning every sample its unique sample identification number (Reference #).

2.1.4 Office Staff are responsible for the initial receipt of samples at the laboratory, the proper completion of the C-of-C, and the entry of analytical requests into the Laboratory Information Management System (LIMS). Office staff is also responsible for the generation of the final report and ensuring the proper delivery of all analytical results to the client.

2.1.5 The Laboratory Manager coordinates with clients and analysts to develop work schedules to meet analytical demands. The Laboratory Manager is available to analysts to assist in analytical troubleshooting. The Laboratory Manager reports directly to the Laboratory Director.

2.1.6 The Quality Assurance Officer reviews the QA/QC results of individual analyses, compiles the data, and reviews the data with the Laboratory Director. The Q.A. Officer is also responsible for ensuring that the overall laboratory operations comply with the Laboratory QA/QC Plan. The Q.A. Officer reports to the Laboratory Director, but has access to the Board of Directors

2.1.7 The Laboratory Director has overall responsibility for the technical operation of the laboratory. The Director is responsible for arranging and overseeing the maintenance of all equipment and instrumentation, as well as the physical maintenance of the laboratory. The Director provides supervision to all laboratory personnel to ensure adherence to all lab-documented procedures including the Quality Manual.

2.2 Personnel Qualifications

Endyne, Inc. will hire only the most qualified and capable applicants for any position to be filled. Each new employee must have a combination of education and experience to adequately demonstrate the specific knowledge necessary to perform their assigned duties as well as a general knowledge of the mathematics and good laboratory techniques, which would be required. Endyne sets several conditions to ensure this minimum level of qualification. All individuals hired for analytical positions will have at least three years of course work towards a four-year college degree in a related science discipline or have completed a post-high school laboratory analyst program at a recognized technical school. Any individual without a college level education would require two years of on the job training in a laboratory environment prior to performing analytical work.

2.3 General Procedural and Ethical Personnel Training

Once hired a new employee will undergo a thorough training regimen to ensure they are capable of performing their tasks to the defined level of acceptance. Proper training consists of an initial introduction to laboratory procedures, continuing general in-house safety and procedural training and where applicable, continuing career development seminars. Most analytical procedures have specific requirements to demonstrate analytical capability. Those requirements are adhered to as well as our general guidelines for training. Documented training activities are stored in employee's personnel files.

2.3.1 Analytical Training

- The trainee reviews literature associated with the published methods. This includes all related internal SOPs such as the method SOP, equipment SOPs such as balance, glassware or pipettor SOPs and, if pertinent, the chromatography integration SOP.
- The trainee also reviews all MSDSs for chemicals used during the procedure. . Analysts must read and become familiar with all elements of each chemical's MSDS, making special awareness of the symptoms of exposure and potential routes of entry.
- If it's a new procedure and there is no internal method SOP, then the analyst reads the original method, writes a summary and reviews the procedure with the Laboratory Manager.
- The trainee works several days under the supervision of an analyst, experienced with the method. This training includes any specific laboratory techniques such as pipetting, syringe use, titration. Equipment maintenance and use such as probes, scales, meters or chromatograph operation.
- The trainee may assist in the generation of data in a trainee mode. However, the trainee may not work independently until certification is fully documented.
- The trainee performs a Demonstration of Capability (DOC). This is an analysis of four replicate quality control samples. The average of the results must be within 20% of the industry-standard expected mean value and the standard deviation must be less than 20%, unless the method specifically indicates otherwise.

- The new analyst completes a DOC Certificate (see Appendix A) which documents his training and proficiency in a method.
- The Laboratory Manager will authorize an analyst to perform a method only after successful completion of the DOC and upon conference with the primary trainer. Documentation of this authorization will be kept in the employee's personnel file in the form of an "Analyst Certification Form".

2.3.2 Initial Training

- An overall review of sample flow through the laboratory is provided, so that they may better understand their relationship within the laboratory.
- A review of safety protocols and emergency response procedures is performed.
- A review of general laboratory procedures, including balance, pipetting, etc. techniques is performed.
- Ethical and legal responsibilities are outlined, including potential punishments and penalties for improper, unethical or illegal actions. Refer to Appendix B.
- A review of Endyne's client confidentiality policy is performed, including the privacy of general client information as well as the delivery of analytical data via phone or electronic means. Refer to Appendix C.

2.3.2.1 Continued Training

- Weekly Inorganic and Organic department meetings are held to discuss safety and quality control issues that arise during the course of the previous week.
- Laboratory wide staff meetings occur as needs dictate. Topics range from employee benefits and staffing changes to analytical issues and ethical/legal responsibilities.
- Employees attend off-site training seminars, which are deemed valuable by management. These have included office skills, hazardous waste handling, safety training, and analytical instrumentation courses.

3.0 Laboratory Environment

Endyne, Inc. consists of three levels covering approximately 3,500 square feet of work area. The top floor consists of meeting rooms and organic prep/extraction lab on one side of the building, on the other side of the building is the microbiology lab and Semi-volatile GC laboratory. The middle floor consists of office space and the Volatile Organics instrumentation lab. The bottom floor consists of the Metals and Inorganics work areas.

Endyne has no equipment requiring extremely tight environmental conditions. Each floor contains its own heating and cooling systems designed to keep temperatures around 70°0 F and humidity around 40%. Electronic scales are mounted on vibration inhibiting platforms. Exhaust hoods are located on each floor. They are inspected and calibrated on an annual basis

Biological work areas are sterilized between uses. Test areas of incompatible activities are separated to eliminate contamination. Work areas are wiped down at the end of each day and good housekeeping practices in general are followed to minimize contamination and health risks.

4.0 Document Control

The Document Control System is designed so that documents, which are generated by the laboratory and deemed critical to proper laboratory operation, are tracked and access is limited to responsible parties. This system ensures that only the most recent revisions are available to the appropriate personnel, and all documents in use have the proper authorization. Documents covered by this system are listed in the Master Document Control List, which lists all the records, their location, and revision history. Records covered in the Document Control List include the following;

- -The Master Document Control List
- -This Quality Assurance Manual
- -All Analytical SOPs
- -Equipment Maintenance SOPs, i.e. Analytical Balance and Pipette SOPs
- -Externally produced literature used in method Development and Troubleshooting.
- -General Laboratory Procedure SOPs, i.e. Mailing and Billing procedures
- -Sampling Procedures, i.e. Coliform Bacteria
- -Employee Qualification Summaries

The Quality Assurance Officer is responsible for maintaining the Document Control System. Master List Controlled Documents are indicated by the paper color indicated in the footer. Uncontrolled copies are determined by being printed on any other colored paper. The Q.A. Officer has control over the supply of the specified colored paper. The Laboratory Director approves all newly released control documents. The Q.A. Officer is responsible for archiving the obsolete documents.

5.0 Sample Management

Endyne has policies and personnel to manage the sample load generated from its clients. In the event that there is a surplus of work so that Endyne could not process all samples within stated hold times, Endyne would follow the protocols as outlined in the Sample Custody SOP for sub-contracting the excess work.

5.1 Review of all New Work

All new work is defined as analytical procedures for which Endyne does not already have an internal documented procedure with established quality control objectives. A list of established analyses is provided in Table 2. All new work is accepted by the Laboratory Director or Laboratory Manager, after ensuring the laboratory has the proper facilities and equipment to perform the analyses. Staff, involved in the new work, meets to discuss any pertinent issues regarding quality assurance considerations.

The plan for any new testing must be reviewed and approved by the Laboratory Director. The designated employee(s) shall write a SOP for the procedure, which will be included in the document control. A DOC will be kept in the employees personnel file.

5.2 Sampling Protocol

In general, samples received by Endyne are collected by representatives of firms contracting with the laboratory to perform the analysis. These firms are responsible for insuring that proper sampling procedures are followed. Endyne's responsibility is limited to providing written sampling procedures for sample collection, preservation, field filtration and transport for specific analysis when requested. Upon request, Endyne can provide expert sampling services for air, water, and solid media.

5.3 Sample Receipt Protocol

Upon arrival at the laboratory, samples and associated Chain-of-Custody (C-of-C) are reviewed for accuracy and sample integrity. In summary, the C-of-C should contain the following information: (For a more detailed review, refer to the Sample Custody SOP)

1. Client and contact person.
2. Project Name
3. Client sample identification (station location or number).
4. Name of sample collector, date and time of collection.
5. Analyses requested.
6. Container type and volume.
7. Type of preservation performed in the field.
8. Special requests or instructions (e.g. rush analyses).
9. Signature of Endyne personnel who received samples.
10. Date and time of receipt.

Samples are then inspected for general condition, which includes the following:

1. If seals are present on containers, an inspection of each seal is made to insure that each seal is intact.
2. Proper sampling containers have been utilized for the requested analyses, and adequate sample volume is available.
3. For volatile organic compounds (VOC), any air bubbles will be noted on the C.O.C.
4. Proper temperature and preservation guidelines have been followed.
5. Comparison of notations on sample containers with those on the Chain-of-Custody (sample location, date, time) are made to insure that all samples which were collected have arrived at the laboratory, and are properly labeled.

If discrepancies or omissions exist in any of these areas, the Laboratory Director and client are notified. If the client cannot be reached, the samples are placed in cold storage (4°C) and no further action is taken until the problem is resolved.

5.3 Sample Receipt Protocol (cont.)

In conjunction with the C-of-C, Endyne documents pertinent sample preservation information and sample condition on the Log-In report. The person receiving the sample records the condition of the samples, the temperature, the accuracy of the C-of-C and the means of delivery to the laboratory. The person prepping the sample records any preservation or sample preparation procedures necessary for sample storage until the time of analysis.

Once logged in, the samples are delivered to the appropriate department, preserved, as appropriate, and/or refrigerated at 4°C.

For further information refer to the SampleCustody SOP.

5.4 Chain-of-Custody

The purpose of the Chain-of-Custody form is to provide a written record, which traces the collection, possession, and handling of samples from the moment of collection to arrival at the laboratory. An example of our Chain-of-Custody Record is provided in Figure 1. The Chain-of-Custody form is signed by the sample collector, by those responsible for the transportation of the sample, and finally, by laboratory personnel. Any breach in the Chain-of-Custody is reported to the Laboratory Director, the sampler, and the client. In most instances, the Chain-of-Custody form will also be used by the client as an order form to specify the requested analyses, and as a reporting sheet for field results. Upon receipt at the laboratory, a carbon copy of the Chain-of-Custody form is returned to the sampler. All original forms become property of the laboratory.

5.5 Sample Preservation

Sample preservation is divided into two areas. They are the following:

1. Field preservation by sampler.
2. Preservation by Endyne personnel.

Endyne, Inc. takes no responsibility for preservation procedures or materials used in the field. As stated in Section 5.2, we will provide methods, bottles and chemicals appropriate for proper field preservation. Any observed deviation of field preservation from these recommended methods will be reported to the client and noted on the Chain of Custody.

If the sample is received without preservation, Endyne personnel will preserve the sample following Table 1, as long as handling the sample will not affect data integrity. Any non-standard preservation requested by the sampler will be reported to the client and noted on the Chain-of-Custody.

5.6 Analytical Holding Times

Also listed on Table 1 are recommended holding times for all analyses. As discussed in Section 5.3, notification will be made to the Laboratory Director of any samples received by Endyne, which have exceeded the specified holding time limit. At Endyne, all samples will be processed within the recommended holding time. If, due to equipment malfunction or other circumstances beyond the control of the laboratory, an analysis cannot be performed at Endyne, a subcontracting laboratory will be assigned to perform the analysis. The client will be informed in writing of Endyne's intention to sub-contract the analysis. Once permission from the client has been granted, a Chain-of-Custody record will be forwarded with all samples sent to the subcontracting laboratory.

5.7 Minimum Required Volumes

Table 1 indicates the minimum required volumes to perform all analyses. During laboratory preservation procedures (see Section 5.5), the sample will be split, if necessary, such that appropriate volumes are preserved for all requested analyses. The notification procedure set forth in Section 5.3 will be implemented for samples with inadequate volume. In these cases, it will be the client's responsibility to decide which analyses to perform.

6.0 Instrumentation and Equipment

The proper operation of the analytical instrumentation, all measuring devices, the de-ionized water supply, as well as the cleanliness of the glassware must be insured to meet the quality control requirements of the laboratory. Individual Standard Operating Procedures are maintained and adhered to for each type of Equipment. The following sections are summaries of those SOPs. A listing of all major pieces of equipment is provided in Table 3.

6.1 De-Ionized Water

A continuous resistivity meter is mounted on the de-ionized water unit. De-ionized water has an acceptable quality when the resistance value ranges from 14 to 18 μohms. If the resistance falls below this value, new filter elements are installed. De-ionized water from the conditioned device is not used until it can pass the 14 to 18 μohms standard. Grab samples are also analyzed using a separate electronic conductivity meter on a monthly basis, to verify the accuracy of the continuous recording device. This conductivity measurement must be < 2.0μmhos.

6.2 Balances, Thermometers and Pipettors

All weights and calibration thermometers are calibrated against NIST traceable standards at an offsite certified facility. In-house use thermometers are calibrated against the off-site calibrated thermometer on an annual basis. Balances are inspected by a certified specialist on an annual basis and an internal calibration is performed at a minimum of once a week. Balances are checked using NIST traceable weights on a daily basis. A variety of pipettors capable of measuring from 10ul to 10ml are calibrated on a monthly basis and spot checked by analysts on a more frequent basis.

6.3 Glassware

All glassware used for measurement is Class A certified. Glassware is cleaned thoroughly with soap and water. Tap water is used for the preliminary wash followed by rinses with de-ionized water. For trace metal analysis, the glassware is also subjected to an acid wash. All glassware used for organic extraction is rinsed with appropriate solvent prior to analysis.

6.4 Instrumentation

Each major piece of instrumentation is assigned to an individual analyst, for which they are responsible. A separate bound notebook or excel spreadsheet is maintained at each instrument station. In this notebook, the daily operation status of the instrument is noted as well as its complete maintenance history. All changes and servicing to the original factory equipment are noted, dated, and signed by the analyst responsible for the instrument. The Laboratory Director is notified of any problems with the equipment. Routine checks of the instrument log notebooks are made by the Laboratory Director to insure proper reporting.

Regular site visits are made to Endyne by manufacturer's service staff for equipment servicing. Some major instruments are covered by service contracts, which provide for rapid response by the manufacturer in the event of an equipment malfunction.

7.0 Internal Quality Control of Test Procedures

All method-referenced analyses performed at Endyne adhere to the quality control standards outlined in the published literature. Methods, which do not specifically reference quality control parameters, as well as in-house developed methods, adhere to, at the minimum, the QA/QC protocols outlined here. In some cases however, some of the following protocols are not possible to perform. Individual method SOPs have the appropriate QA/QC protocols referenced within them.
Endyne's internal QA/QC plan consists of method and analyst capability demonstrations, as well as a suite of quality control protocol steps during the analysis of samples.

7.1 Method and Analyst Capability Demonstrations

All records include references to the raw data files or dates analyzed, so that calculations can be regenerated from the original data.
Demonstration of Capability (DOC)- Every new analyst performs a set of four mid-range concentration LFBs. The LFBs should be prepared as one sample, then split into four aliquots. The percent standard deviation and average percent recovery are determined for each analyte. Each must be less than 20% to ensure proper capability of the analyst for that method. Documentation of the DOC is kept in the analyst's personnel file.
Method Detection Limit Study (MDL)- The analyst performs a set of seven LFBs at a concentration of 2 to 5 times the expected MDL value. The standard deviation is multiplied by 3.14 to determine the laboratories MDL. The MDL should be less than half the laboratory's reporting limit. MDLs are performed on an annual basis at the beginning of each year. Copies of the MDL are stored with the QA/QC data for the method.

7.2 Quality Control Protocols

Endyne generates control limits for the quality control samples using method-defined limits. Typically, those limits are +/- three standard deviations of the average recovery. In the event there is no accrued history of QC data an absolute acceptance limit of 70%-130% is employed. Duplicate precision limits are based on 3.27 multiplied by the mean relative percent differences. In the event, there is no accrued history of duplicate data an absolute acceptance limit of 20% relative percent difference is applied.

Analytical Calibration Line- A minimum of a 3-point calibration line not including "0", which must have a linear correlation of 0.995 or better. The lowest point in the line must be less than the reporting limit or regulatory level.
Continuing Calibration Check (CCC)- A calibration line standard is analyzed if a calibration line already exists. A CCC is analyzed at the start of each analytical day.
Laboratory Reagent Blank (LRB)- A reagent water sample which is treated as a normal sample and must be shown to be free of all target analytes at reportable levels. An LRB is run with every 20 samples or each analytical batch, whichever is more frequent.
Laboratory Fortified Blank (LFB)- An independent source standard containing all the target compounds is analyzed like a normal sample. A LFB is run with every 20 samples or each analytical batch, whichever is more frequent.
Matrix Duplicate (MD)- Aliquots of the same sample which are analyzed identically. A MD is run with every 20 samples or each analytical batch, whichever is more frequent
Matrix Spike (MS)- A sample aliquot to which a known amount of analyte(s) is added. A MS is run with every 20 samples or each analytical batch, whichever is more frequent.
Matrix Spike/Duplicate (MS/D)- This quality control parameter may be employed in place of the Sample Duplicate and Matrix Spike. Two sample aliquots are spiked with known amounts of analyte and the percent recovery and percent difference is determined for the samples.

7.3 Analytical Standards

Endyne takes special care to ensure the accuracy of all analytical standards. Standards are made in-house from neat materials or purchased from suppliers with at least an ISO 9001 certification. All Certificates of Analysis from suppliers are kept on file in the department in which they are used. In-house standards are made using Class A glassware and a NIST traceable balance. All working standards are documented in individual method logbooks that insure traceability to the original manufacturer.

7.4 Data Storage and Archival

All raw data and calculations, sufficient to completely reconstruct the data packet, are kept on file by the laboratory for a period of 5 years. New York drinking water analyses are kept on file for 10 years. Data storage during the course of the year is outlined in individual SOPs. At the start of each calendar year all data and paperwork is archived in banker boxes. These boxes for the two previous years are kept on site. Data older than two years is kept off-site in a secured location. In case of transfer of ownership or closure of business, the owners of Endyne will continue storage of required documents for the required time or ensure transfer of documents to new ownership.

7.5 Internal Audit and Data Review

-Raw Data - Each analyst must keep detailed information of the raw data in a bound notebook or on the computer printout. Entries must include the analyst initials, date and time of analysis, sample identification number, parameter tested and analytical method, documentation of all readings, and results of all weightings and dilutions. All calculations documenting the final concentration of the constituent as well as determinations of all quality control samples must be recorded with the data.

-Data Checking – Data checking is initially performed by an independent analyst trained in the analytical methodology. After the analyst has processed all the associated data and recorded the results of an analysis, the independent analyst reviews the raw data and associated quality control samples. The reviewer then signs off on the Log-In report form indicating that they have fully reviewed the work. The independent analyst is held equally accountable for certifying all quality control aspects of the methodology. The completed report is submitted to the Laboratory Director for final review and approval before being released to the client.

-System Audits – The QA Officer and/or the Laboratory Director will review individual methods on an annual basis with the primary analyst. Procedures will be compared to the SOP and Quality Systems Manual to confirm adherence to both. Any corrective actions will be documented. Where audit findings cast doubt on the validity of results, clients will be contacted immediately in writing.

-Managerial Review – On an annual basis, the Laboratory Director will review the quality system and its testing and calibration activities. The review will be based on feedback from the QA Officer, the results of proficiency tests, as well as audits from external certification officers. Any necessary changes will be implemented at this time and documented in the Quality Systems Manual.

-Internal Audit – On an annual basis, the Quality Control Officer will present to the management staff a summary of the Internal Audit. This will include the findings of the systems audits as well as the managerial review, proficiency testing results and customer complaints. The outline of this process is defined in the Internal Audit SOP.

8.0 Testing Discrepancies and Corrective Action

Occasionally analytical data fails the quality control protocols outlined in each method SOP. Procedures for determining the source of these failures are outlined in the SOP. In general, the primary analyst immediately reviews the work and assesses where the error occurred, corrects the deficiency and reanalyzes the associated samples. No further sample analysis is performed until the source of the error is determined and corrected. The laboratory manager and director are available for immediate consultation if necessary. In some instances, due to the sample volume submitted or the expiration of the analytical holding time, it is impossible to reanalyze the sample within QA/QC protocols. In that event the client is notified of the violation by telephone or in writing. No data, which is outside method specified quality control parameters, will be reported without the appropriate notation informing the client of the transgression.

9.0 Exceptionally Permitted Departures from Documented Policies

The Laboratory Director is responsible for ensuring adherence to the documented policies of the laboratory. However, in some instances departure from documented procedure may be necessary. In this instance, digression from the procedure is allowed as long as the changes are fully documented as well as the reason why the deviation occurred and the Laboratory Director approves all the departures.

10.0 Proficiency Testing and Certification

Endyne participates in several proficiency-testing programs for drinking water, wastewater and air. Endyne participates in a Water Supply testing program on a semi-annual basis. The testing encompasses all of the parameters for which Endyne receives certification for from the State of Vermont. Endyne also participates in semi-annual wastewater proficiency testing administered by the New York State Environmental Laboratory Accreditation Program (ELAP). Both sets of proficiency testing are approved by the National Environmental Laboratory Accreditation Council (NELAC). Endyne also participates quarterly in a suite of testing for in-door air contaminants. These tests are administered by the American Industrial Hygienists Association (AIHA). An addendum to this manual is a current listing of State of Vermont and NELAC certified methods and parameters.

11.0 Complaints and Corrective Action Procedures

Endyne continually strives to improve data quality and customer satisfaction. In an effort to track complaints and quality control problems, Endyne maintains a file where complaints, from clients or other parties, and their corrective actions are recorded. A five-step plan is implemented to correct the deficiencies.
Endyne documents Corrective Actions using a standardized investigative Report Template (Corrective Action Report). Initially the complaint, whether from a client or discovered in any of the internal QA review processes, is documented. Assessment of the route cause of the problem is performed. A program to address the cause is implemented. Initial assessment to determine if the problem/complaint is corrected. Finally the system is reevaluated at a later date to ensure the proposed solution is still effective.

12.0 Reporting of Results

Endyne policy is to report results solely to the client listed on the Chain-of-Custody. Delivery of results is accomplished in four ways. The only authoritative results reported by Endyne are in the form of a hard copy report with the original signature of the Laboratory Director or appointed signer. The other forms of data deliverables are by fax ,e–mail or verbal. Clients who request a verbal transmission of results must concretely establish themselves as the proper client, This is accomplished by referencing the C-of-C number in the upper right hand corner. Clients who request a fax must indicate on the C-of-C whom will receive results or if the request is made by phone proof must be made that they are the stated client. This is usually accomplished by referencing the distinctive C-of-C sequence number. Deliverables in the form of e-mails are only provided to established customers, whom have made formal arrangements for receipt of results. All electronic transmissions are accompanied with a disclaimer stating that the material is confidential information intended solely for the recipient and to notify Endyne immediately upon incorrect receipt. Refer to Appendix C.

13.0 Revision History

Revision 1:	July 18, 2000:	Complete Draft
Revision 2:	March 15, 2001:	Minor Revision
Revision 3:	March 15, 2002:	Minor Revision
Revision 4:	January 28, 2003:	Minor Revision
Revision 5:	February 28, 2003:	Added Certification Addendum
Revision 6:	March 2, 2004:	2.2 Personnel Qualifications 2.3.1 Analytical Training 7.5 Internal Audit and Data Review
Revision 7: September 20, 2005		2.0 Added reference to Scitest, Inc. in corporate Structure. 3.0 Laboratory Environment- Noted reconstruction & location of departments. 4.0 Add instrument manuals to Document Control 11.0 Multi-point Corrective Action Report

14.0 References

Standard Methods for the Examination of Water and Wastewater. 17th Edition. APHA, AWWA, WPCF. Denver Colorado (1989)

Test Methods for Evaluating Solid Watse, Physical/Chemical Methods (SW-846 Version 2) on CD-ROM. NTIS U.S. DOC. Springfield, VA (1997).

EPA Methods and Guidance for Analysis of Water (Version 2) on CD-ROM. Office of Science and Technology, U.S. EPA. Washington DC (1999)

Environmental Laboratory Approval Program Certification Manual. New York Department of Health. Albany, NY (1999)

1999 Annual New York and Pennsylvania Environmental Laboratory Conference & Exposition. August 8th-10th, 1999.

NELAC Quality Systems Checklist Rev. 6. National Environmental Laboratory Accreditation Conference. 1997.

Endyne, Inc.
160 James Brown Dr.
Williston, VT 05495

ENDYNE, INC.
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CHAIN-OF-CUSTODY-RECORD

37472

Project Name:		Reporting Address:	Billing Address:
Endyne Order ID: (Lab Use Only)	-O -I -S	Company: Contact Name/Phone #:	Sampler Name: Phone #:

Ref # (Lab Use Only)	Sample Identification	Matrix	GRAB	COMP	Date/Time	Sample Containers No.	Sample Containers Type/Size	Field Results/Remarks	Analysis Required	Sample Preservation	Rush

Relinquished by:	Date/Time	Received by:	Date/Time	Received by:	Date/Time

New York State Project: Yes ____ No ____

Requested Analyses

1	pH	6	TKN	11	Total Solids	16	Sulfate	21	1664 TPH/FOG	26	8270 PAH
2	Chloride	7	Total P	12	TSS	17	Coliform (Specify)	22	8015 GRO	27	PP13 Metals
3	Ammonia N	8	Total Diss. P	13	TDS	18	COD	23	8015 DRO	28	RCRA8 Metals
4	Nitrite N	9	BOD	14	Turbidity	19	8021B	24	8260/8260B	29	
5	Nitrate N	10	Alkalinity	15	Conductivity	20	8010/8020	25	8270 B/N or Acid	30	
31	Metals (As Is, Total, Diss.) Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sr, Ti, Tl, V, Zn										
32	TCLP (Specify: volatiles, semi-volatiles, metals, pesticides, herbicides)					33					
34	Other										

(White, Yellow, Pink Copy - Laboratory / Goldenrod Copy - Client)

Document Control Copy on Buff colored paper. Any other color is not official version

TABLE 1
Recommendation for Sampling and Preservation of Samples According to Measurement

Measurement	EPA Method	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
100 PHYSICAL PROPERTIES					
Color	110.3	100	P,G	Cool, 4°C	48 hrs.
Conductance	120.1	100	P,G	Cool, 4°C	28 days
Hardness	6010	100	P,G	HNO_3 to pH <2	6 mos.
Odor	140.1	500	G only	Cool, 4°C	24 hrs.
pH	150.1	25	P,G	None Required	Analyze Immediately
Residue/Solids:					
Filterable	160.1	250	P,G	Cool, 4°C	7 days
Non-filterable	160.2	250	P,G	Cool, 4°C	7 days
Total	160.3	250	P,G	Cool, 4°C	7 days
Volatile	160.4	250	P,G	Cool, 4°C	7 days
Settleable Matter	160.5	1000	P,G	Cool, 4°C	48 hrs.
Temperature	170.1	100	P,G	None Required	Analyze Immediately
Turbidity	180.1	100	P,G	Cool, 4°C	48 hrs.
200 METALS					
Dissolved	3113B/200.7/6010	500	P,G	Filter on-site HNO_3 to pH <2	6 mos.
Suspended	3113B/200.7/6010	500	P,G	Filter on-site	6 mos.
Total	3113B/200.7/6010	500	P,G	HNO_3 to pH <2	6 mos.
Chromium^{+6}	7196A	200	P,G	Cool, 4°C	24 hrs.
Soils	SM3113B/EPA6010	50grams	P,G	Cool, 4°C	6 mos.

TABLE 1
Recommendation for Sampling and Preservation of Samples According to Measurement

Measurement	EPA Method	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
300 INORGANICS, NON-METALLICS					
Acidity	305.1	100	P,G	Cool, 4°C	14 days
Alkalinity	310.1	100	P,G	Cool, 4°C	14 days
Bromide	300.0	100	P,G	None Required	28 days
Chloride	300.0	50	P,G	None Required	28 days
Chlorine	330.5	200	P,G	None Required	Analyze Immediately
.Cyanides	335.1	250	G	Cool, 4°C NaOH of pH >12 0.6 g ascorbic acid	14 days
Fluoride	340.2	200	P,G	None Required	28 days
Nitrogen:					
Ammonia	350.3	100	P,G	Cool, 4°C H_2SO_4 to pH <2	28 days
Kjeldahl, Total	351.4	100	P,G	Cool, 4°C H_2SO_4 to pH <2	28 days
Nitrate	300.0	50	P,G	Cool, 4°C	48 hours
Nitrite	300.0	50	P,G	Cool, 4°C	48 hours
Phosphorus:					
Ortho-Phosphate,	365.2	100	G	Cool, 4°C H_2SO_4 to pH <2	28 days.
Dissolved	365.2	100	G	Filter,Cool, 4°C H2SO4 to pH <2	28 days
Total	365.2	150	G	Cool, 4°C H_2SO_4 to pH <2	28 days
Total Dissolved	365.2	50	G	Filter on-site Cool, 4°C H_2SO_4 to pH <2	24 hrs.

TABLE 1
Recommendation for Sampling and Preservation of Samples According to Measurement

Measurement	EPA Method	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
300 INORGANICS, NON-METALLICS					
Sulfate	300.0	50	P,G	Cool, 4°C	28 days
Sulfide	376.1	500	P,G	Cool, 4°C Add 2ml Zinc Acetate and NaOH to pH >9	2 days
Sulfite	377.1	50	P,G	None Required	Analyze Immediately
Radioactivity:					
Gross Alpha	Rad TSU	2000	P	None	28days
Radium 226,228	Rad TSV	4000	P	None	28 days
400 ORGANICS					
BOD	405.1	1000	P,G	Cool, 4°C	24 hrs.
BOD, Soluble	405.1	1000	P,G	Cool, 4°C	24 hrs
COD	410.1	50	P,G	Cool, 4°C H_2SO_4 to pH <2	28 days
Organic Carbon	415.1	25	P,G	Cool, 4°C H_2SO_4 or HCl to pH <2	28 days
Phenolics	420.1	500	G only	Cool, 4°C H_2SO_4 to pH <2	28 days
MBAs	425.1	1000	P,G	Cool, 4°C	48 hrs.
Oil & Grease	1664	1000	G only	Cool, 4°C H_2SO_4 to pH <2	28 days

TABLE 1
Recommendation for Sampling and Preservation of Samples According to Measurement

Measurement	EPA Method	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
500 SERIES Drinking Water					
Volatiles	504.1	2 40-ml vials	G only	Cool, 4°C 10 mg $Na_2S_2O_3$	14 days
Pesticides/PCB	505	2 40-ml vials	G only	Cool, 4°C 10 mg $Na_2S_2O_3$	14 days/
Herbicides	515	2 1000-ml	Amber G	4°C 5% $Na_2S_2O_3$ 1:1 HCl	14 days/ 14 days
S.O.C.	525	2 1000-ml	Amber G	4°C 5% $NaSO_3$ 1:1 HCl	14 days/ 14 days
Carbamates	531	2 40ml vials	G only	Cool, 4°C 1.8mls MCA	28 days
V.O.C	524.2 503/502	2 40ml vials	G only	Cool, 4°C Ascorbic Acid 1:1 HCl	14 days
8000 SOLIDS					
Volatiles	8010/8021B 8260/8260B	Full 40 ml vial	G only	4°C	14 days
Pesticides	8081A	250 ml	Amber G	4°C	14 days/ 40 days
PCBs	8082	250 ml	Amber G	4°C	14 days/ 40 days
S.V.O.C. BN/A	8270C	250 ml	Amber G	4°C	14 days/ 40 days
TPH GRO	8015B	2 40-ml	G only	4°C	14 days
TPH DRO	8015B	2 40-ml	G only	4°C	14 days 40 days

TABLE 1
Recommendation for Sampling and Preservation of Samples According to Measurement

Measurement	EPA Method	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
8000 WATER					
Volatiles	8010/8020 8260/8260B	2 40-ml vials	G only	No bubbles 4°C HCl to pH <2	14 days
Pesticides	8081A	2 1000-ml	Amber G	4°C pH between 5-9	7 days/ 40 days
PCBs	8082	2 1000ml	Amber G	4°C pH between 5-9	7 days/ 40 days
S.V.O.C. BN/A	8270C	2 1000-ml	Amber G	4°C	7 days/ 40 days
TPH GRO	8015B	2 40-ml	G only	No bubbles 4°C HCl to pH <2	14 days
TPH DRO	8015B	2 40-ml	G only	No bubbles 4°C HCl to pH <2	7 days
MICROBIOLOGICAL					
Total and Fecal Coliform	Not Applicable	100 ml	125 ml Sterile Plastic	4°C 0.008% Na2s203	30 hrs (24 hrs, recommended)
Escherichia coli	Not Applicable	100 ml	125 ml Sterile Plastic	4°C	24 hrs
Heterotrophic	Not Applicable	100 ml	125 ml Sterile Plastic	4°C	8 hrs

TABLE 1 FOOTNOTES

1. Referenced Methods are from approved publications from Standard Methods 17th Edition, Solid Waste 846 and Environmental Protection Agency

2. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, pgs. 72-82 (1976) Method D-3370.

3. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (teflon liner is preferred.

4. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

5. When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFS Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.15 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); Sodium hydroxide (NaOH) in water solutions at concentrations or 0.080% by weight or less (pH about 12.3 or less).

6. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee , or monitoring laboratories, has data on file to show that the specific types of sample under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

7. Should only be used in the presence of residual chlorine. The sample is filtered and the NaOH is added to pH 12.

8. For samples from non-chlorinated drinking water supplies, concentrated H_2SO_4 should be added to lower sample pH to less than 2. The sample should be analyzed before 14 days.

TABLE 2 Analytical Methods
Organic Analyses

ANALYTE	EPA METHODS
Volatile Organic Compounds	
VOCs by GC/MS	8260/8260B
GC/MS Calibrated Nontarget Compounds	8260B
SDWA Drinking Water Volatiles	524.1
Purgeable Aromatics by GC/PID	8020/8021B
Purgeable Halocarbons by GC/MS	8010
Combined Purgeables by GC/MS	8010&8020
SDWA EDP, DBCP by GC/ECD	504.1
Base Neutral/Acid Extractables	8270C
Acid Extractables	8270C
Base Neutral Extractables	8270C
Polynuclear Aromatics	8270C
Organochlorine Pesticides & PCBs	8081A
PCBs	8082
Herbicides	8150
SDWA Pesticides	505/525.2
SDWA PCBs	505
SDWA Herbicides	515.2
SDWA Carbamates*	531.1
Anti-Freeze	8015B
TPH-GRO	8015B
TPH-DRO	8015B/1664
Petroleum Hydrocarbon Identification	GC/FID
Oil&Grease	EPA 1664
Oil and Grease by IR	EPA 413.2
Petroleum Hydrocarbons by IR	EPA 418.1

TABLE 2 Analytical Methods
Inorganic Analyses

PARAMETER	EPA METHODS
Alkalinity	EPA 310.1
Biological Oxygen Demand	EPA 405.1
Carbon *:	
Total Inorganic* (water)	EPA 415.1
Total Organic* (water)	EPA 415.1
Total* (soil)	EPA415.1
Total Organic* (soil)	EPA415.1
Chemical Oxygen Demand	EPA 410.2
Chloride	EPA 325.1
Chlorine	EPA 330.5
Color	EPA 110.3
Cyanide * - Total	EPA 335.2
Cyanide * - Total & Amenable	EPA 335.1
Fluoride	EPA 340.2
Hardness	SM314A
Halides, Total Organic (TOX)*:	
Water *	SW 9020
Solids *	9020
Lime Requirement	----
Nitrogen	
Ammonia	350.1
Nitrate	300.0
Nitrite	300.0
Total Kjedahl	351.2

TABLE 2 Analytical Methods
Inorganic Analyses

PARAMETER	EPA METHODS
pH	150.1
Phenols - Total	420.3
Phosphorus:	
Total	365.1
Ortho	365.1
Total Dissolved	365.1
Solids	
Total	160.3
Percent	160.3
Suspended	160.2
Dissolved	160.1
Volatile	160.4
Suspended Volatile	160.2/160.4
Settleable	160.5
Specific Conductance	120.1
Sulfate	300.0
Sulfide	376.1
Sulfite	377.1
Surfactants - MBAs	425.1
Turbidity	180.1

TABLE 2 Analytical Methods
Metal Analyses

ANALYTE	EPA METHODS
Aluminum	200.7/6010
Antimony	200.7/6010
Arsenic	200.7/3113B
Barium	200.7/6010
Beryllium	200.7/6010
Boron	200.7/6010
Cadmium	200.7/3113B
Calcium	200.7/6010
Chromium	200.7/6010
Colbalt	200.7/6010
Copper	200.7/6010
Iron	200.7/6010
Lead	200.7/3113B
Magnesium	200.7/6010
Manganese	200.7/6010
Mercury (cold vapor)	245.1/7470
Molybdenum	200.7/6010
Nickel	200.7/6010
Potassium	200.7/6010
Selenium	200.7/3113B
Silver	200.7/6010
Sodium	200.7/6010

TABLE 2 Analytical Methods
Metal Analyses

Thallium	200.7/3113B
Vanadium	200.7/6010
Zinc	200.7/6010
Digestions:	
Water	3020
Soil	3050

Footnotes

Asterisk (*) indicates analysis performed by a sub-contracted laboratory

TABLE 3
Laboratory Equipment and Instrumentation

Item	No. of Units	Manufacturer	Model
ANALYTICAL BALANCE 0.1 mg sensitivity,Stable base Class S weights NIST Traceable Service contracts	1	Mettler	AD-160
MAGNETIC STIRRER Variable Speed TFE coated stir bar	3	Corning	PC 351
pH METER ± 0.05 units, Readability ± 0.1 units Line or battery, Usable with specific ion electrodes	1	Orion	940
CONDUCTIVITY METER Readable in ohms or mhos Range of 2 ohms to 2 megaohms, Line or battery	1	YSI	35
HOT PLATE Temperature control	6	Fisher(3) Cimarec(3)	
COLOR STANDARDS To verify wavelengths on photometers Should cover 200 to800 nm	1	Oxford	spectro-check
REFRIGERATOR Sample Storage	3	United	
REFRIGERATOR Standards Storage	4	Avanti, etc.	
DRYING OVEN Gravity or convention, Controlled from room Temperature to 400°C for cleaning organic glass	2	VWR	1305U
THERMOMETERS Mercury-filled celcius, 1°C or finer subdivision to 180°C Certified by or traceable to NIST	15		
GLASSWARE Borosilicate Class A volumetric			

TABLE 3
Laboratory Equipment and Instrumentation

Item	No. of Units	Manufacturer	Model
SPECTROPHOTOMETER	1	Milton Ray	401
Range 400 to 700 nm	1	Bausch & Lomb	700
Band width not greater than 20 nm			
Use several size and shape cells			
Path length 1 to 5 cm			
SPECIFIC ION METER	2	Orion	9401
Readable & accurate to ±1 mV			
ELECTRODES	2	pH, F	960
As needed			
INDUCTIVELY COUPLED PLASMA	1	Leeman	PS1000
Computer control, Background coordination			
Radio frequency generator, Argon gas supply			
WATER BATH	1	Precision	Coliform
Electric or steamed heat	1	Precision	185
Heat to 100°C, Controllable with 5°C			
ION CHROMATOGRAPH	1	Waters	HPLC System
U.V. and Conductivity detector, Separator columns			
	1	Dionex	AS-40/DX-120
Conductivity detector/Carbonate eluent			
ATOMIC ABSORPTION SPECTROPHOTOMETER w/Zeeman Correction	1	Varian	AA-20
Single channel, Single or double beam			
Grating monochrometer, Photomultiplier detector			
Adjustable slits, Range 190 to 800 nm			
Readout system:			
PIPETTORS	8	Finntip/Eppendorf	
Microliter capacity with, disposable tips			
Sizes 5 to 10000 microliters			
RO Water Purification System	1	MilliporeWaters	Milli-Q

TABLE 3
Laboratory Equipment and Instrumentation

Item	No. of Units	Manufacturer	Model
GLASSWARE		Wheaton/Kimble	
Separator Funnels			
Kuderna Danish (K-D), Concentrators			
Water bath for K-D			
GAS CHROMATOGRAPHS	3	Hewelett-Packard	5890
±0.2°C oven, Temperature control	3	Hewelett-Packard	5890 Series II
GC Detectors			
Electron Capture Detector	1	Hewelett Packard	
Flame Ionization Detector	1	Hewelett Packard	
	1	OI Analytical	
Photo-ionization Detector	1	HNU	PI-52
	2	OI Analytical	4560
Mass Spectrometer:	1	Hewelett Packard	5970
	1	Hewelett Packard	5971
GC Purge and trap system	2	Tekmar	ALS/LSC2
	1	Tekmar	LSC2000/ALS2016
	2	OI	MPM-16
GC Semi-Vol. Autosampler	2	Hewelett Packard	7673
Dessiccator	4	Kimax	
Muffle furnace	1	Fisher	184 A
Centrifuge	1	Fisher	Centrific
Fluorometer	1	Turner	20-005 R
Automated Colorimeter	1	Lachat	Quickchem 8000

Appendix A

Demonstration of Capability

Certification Statement

Date: _____ Method I.D.: _____

Analyst Name: _____ Matrix: _____
Analyst agrees to use only the most current version of the applicable SOP.

Analyst Signature: _____ DOC Attached (Y / N)

Summary of Training:

_____
_____
_____
_____
_____
_____
_____

We, the undersigned, certify that:

1. The analyst(s) identified above, using the cited test methods, which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program (NELAP), have met the Demonstration of Capability (DOC).

2. The analyst identified on this certification performed the test method

3. A copy of the test method and the laboratory-specific SOPs are available for personnel on-site.

4. The data associated with the DOC are true accurate complete and self-explanatory.

5. All raw data necessary to reconstruct and validate these analyses have been retrained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Harry Locker Ph.D.	_____	_____
Technical Director	Signature	Date
Mark Fausel	_____	_____
Q.A Officer	Signature	Date

Appendix B

Ethical Training Attestation

_____
Analyst Name

I, the undersigned, hereby attest that I have received training in the ethical conduct policy of Endyne, Inc. and that I agree to abide by those policies as outlined below.

1. I understand that Endyne, Inc. considers high quality data to be of paramount importance, and that the confidence of its clients in that data is of critical importance to Endyne's continued success. To that end, Endyne considers falsifying or biasing data to be a material breech in our pledge to our clients.

2. I am free of any commercial, financial and other undue pressures, which might adversely affect the quality of my work. Ex: I receive no compensation from external or internal agents which would induce me to falsify or not qualify what would otherwise be unacceptable data.

3. I understand that unethical behavior includes any means by which I intentionally unduly alter data to put it in a more favorable light. Examples include peak shaving in chromatography, time shifting in regards to holding times. Falsifying analytical data so as to make data more or less beneficial to a client. Falsifying quality control data to make it more acceptable.

4. Endyne does not promote, condone or accept unethical behavior and takes responsibility for ensuring that analysts are aware of consequences for such actions. Repercussions may include punitive actions up to and including immediate dismissal.

5. I understand that other repercussions of unethical behavior can include criminal and civil actions resulting in significant fines and/or jail time.

I have read and agree to adhere to the above statement.

Analyst Signature:_____ Date:_____

Appendix C

Client Confidentiality and Privacy Policy

1. I understand that it is Endyne's policy that our clients have a right to the privacy of their information, in all of its manifestations, and that they are free from the risk of disclosure of this information from any current or past employee of Endyne.
2. I understand item 1. includes any discussions in which the name or identity of a client is revealed to any individual not currently employed by Endyne. The disclosure of this identity includes any reference in which the identity of the client is implicit and that the revelation of this information in any way could demean the standing of that client in the community.
3. I understand that this policy applies not only to our customers but to any projects or work performed by our customers for which the identity of their clients has been made known to us.
4. I have been apprised of Endyne's procedures for the release of all data, be it verbal, electronic or written. I understand the only authoritative results reported by Endyne are in the form of a hard copy report with the original signature of the Laboratory Director or appointed signer.
   a. Clients who request a verbal notification of results must establish proof of identity as the proper client. This is accomplished by referencing the C-of-C number in the upper right hand corner.
   b. Clients who request a fax must indicate on the C-of-C whom will receive results or if the request is made by phone, the individual must prove they are the stated client. This is usually accomplished by referencing the distinctive C-of-C sequence number.
   c. Deliverables in the form of e-mails are only provided to established customers, whom have made formal arrangements for receipt of results.
   d. All electronic transmissions are accompanied with a disclaimer stating that the material is confidential information intended solely for the recipient and to notify Endyne immediately upon incorrect receipt

I acknowledge that I have read this Quality Systems Manual and agree to adhere to the policies outlined within.

Name	Date

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Addendum

Expires 12:01 AM April 01, 2007
Issued April 01, 2006
Revised January 22, 2007

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

DR. HARRY B. LOCKER
ENDYNE INC
160 JAMES BROWN DRIVE
WILLISTON, VT 05495

NY Lab Id No: 11263
EPA Lab Code: VT00024

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Disinfection By-products

Dibromoacetic acid	EPA 552.2
Dichloroacetic acid	EPA 552.2
Monobromoacetic acid	EPA 552.2
Monochloroacetic acid	EPA 552.2
Trichloroacetic acid	EPA 552.2

Drinking Water Bacteriology

Coliform, Total	SM 18-20 9223

Drinking Water Chlorinated Acids

2,4,5-TP (Silvex)	EPA 515.2
2,4-D	EPA 515.2
Dalapon	EPA 515.2
Dicamba	EPA 515.2
Dinoseb	EPA 515.2
Pentachlorophenol	EPA 515.2
Picloram	EPA 515.2

Drinking Water Metals I

Arsenic, Total	SM 18-19 3113B
Barium, Total	EPA 200.7
Cadmium, Total	EPA 200.7
Chromium, Total	EPA 200.7
Copper, Total	EPA 200.7

Drinking Water Metals I

Iron, Total	EPA 200.7
Lead, Total	SM 18-19 3113B
Manganese, Total	EPA 200.7
Mercury, Total	EPA 245.1
Selenium, Total	SM 18-19 3113B
Silver, Total	EPA 200.7
Zinc, Total	EPA 200.7

Drinking Water Metals II

Aluminum, Total	EPA 200.7
Antimony, Total	SM 18-19 3113B
Beryllium, Total	EPA 200.7
Nickel, Total	EPA 200.7
Thallium, Total	EPA 200.9

Drinking Water Metals III

Calcium, Total	EPA 200.7
Magnesium, Total	EPA 200.7

Drinking Water Miscellaneous

Benzo(a)pyrene	EPA 525.2
Bis(2-ethylhexyl) phthalate	EPA 525.2
Butachlor	EPA 525.2
Di (2-ethylhexyl) adipate	EPA 525.2

Serial No.: 31687

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Addendum

Expires 12:01 AM April 01, 2007
Issued April 01, 2006
Revised January 22, 2007

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

DR. HARRY B. LOCKER
ENDYNE INC
160 JAMES BROWN DRIVE
WILLISTON, VT 05495

NY Lab Id No: 11263
EPA Lab Code: VT00024

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Volatile Aromatics

1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

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DR. HARRY B. LOCKER
ENDYNE INC
160 JAMES BROWN DRIVE
WILLISTON, VT 05495

NY Lab Id No: 11263
EPA Lab Code: VT00024

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Amines	
Pyridine	EPA 8270
Bacteriology	
oliform, fecal	SM 18-20 9222D
Benzidines	
3,3' -Dichlorobenzidine	EPA 625
	EPA 8270
Benzidine	EPA 625
	EPA 8270
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 608
	EPA 8081A
4,4'-DDE	EPA 608
	EPA 8081A
Aldrin	EPA 608
	EPA 8081A
alpha-BHC	EPA 608
	EPA 8081A
Chlordane Total	EPA 608
	EPA 8081A
delta-BHC	EPA 608
	EPA 8081A
Chlorinated Hydrocarbon Pesticides	
Dieldrin	EPA 608
	EPA 8081A
Endosulfan I	EPA 608
	EPA 8081A
Endosulfan sulfate	EPA 608
	EPA 8081A
Endrin	EPA 608
	EPA 8081A
Endrin aldehyde	EPA 8081A
Heptachlor	EPA 608
	EPA 8081A
Heptachlor epoxide	EPA 608
	EPA 8081A
Lindane	EPA 608
	EPA 8081A
Methoxychlor	EPA 608
	EPA 8081A
Toxaphene	EPA 608
	EPA 8081A
Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA 625
	EPA 8270

erial No.: 31688

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All approved analytes are listed below:

Nitrosoamines

N-Nitrosodi-n-propylamine	EPA 625
	EPA 8270
-Nitrosodiphenylamine	EPA 625
	EPA 8270

Nutrient

Ammonia (as N)	EPA 350.1
Kjeldahl Nitrogen, Total	EPA 351.2
Nitrate (as N)	EPA 300.0
Nitrite (as N)	EPA 300.0
Phosphorus, Total	EPA 365.1

Phthalate Esters

Benzyl butyl phthalate	EPA 625
	EPA 8270
Bis(2-ethylhexyl) phthalate	EPA 625
	EPA 8270
Diethyl phthalate	EPA 625
	EPA 8270
Dimethyl phthalate	EPA 625
	EPA 8270
Di-n-butyl phthalate	EPA 625
	EPA 8270
Di-n-octyl phthalate	EPA 625

Phthalate Esters

Di-n-octyl phthalate	EPA 8270

Polychlorinated Biphenyls

PCB-1016	EPA 608
	EPA 8082
PCB-1221	EPA 608
	EPA 8082
PCB-1232	EPA 608
	EPA 8082
PCB-1242	EPA 608
	EPA 8082
PCB-1248	EPA 608
	EPA 8082
PCB-1254	EPA 608
	EPA 8082
PCB-1260	EPA 608
	EPA 8082

Polynuclear Aromatics

Acenaphthene	EPA 625
	EPA 8270
Acenaphthylene	EPA 625
	EPA 8270
Anthracene	EPA 625

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All approved analytes are listed below:

Priority Pollutant Phenols

4-Nitrophenol	EPA 8270
Cresols, Total	EPA 8270
Pentachlorophenol	EPA 625
	EPA 8270
Phenol	EPA 625
	EPA 8270

Purgeable Aromatics

1,2-Dichlorobenzene	EPA 624
	EPA 8260B
1,3-Dichlorobenzene	EPA 624
	EPA 8260B
1,4-Dichlorobenzene	EPA 624
	EPA 8260B
Benzene	EPA 624
	EPA 8260B
Chlorobenzene	EPA 624
	EPA 8260B
Ethyl benzene	EPA 624
	EPA 8260B
Toluene	EPA 624
	EPA 8260B
Total Xylenes	EPA 624

Purgeable Aromatics

Total Xylenes	EPA 8260B

Purgeable Halocarbons

1,1,1-Trichloroethane	EPA 624
	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 624
	EPA 8260B
1,1,2-Trichloroethane	EPA 624
	EPA 8260B
1,1-Dichloroethane	EPA 624
	EPA 8260B
1,1-Dichloroethene	EPA 624
	EPA 8260B
1,2-Dichloroethane	EPA 624
	EPA 8260B
1,2-Dichloropropane	EPA 624
	EPA 8260B
2-Chloroethylvinyl ether	EPA 624
	EPA 8260B
Bromodichloromethane	EPA 624
	EPA 8260B
Bromoform	EPA 624
	EPA 8260B

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All approved analytes are listed below:

Wastewater Metals II

Aluminum, Total	EPA 200.7
Antimony, Total	SM 18-19 3113B
Arsenic, Total	SM 18-19 3113B
Beryllium, Total	EPA 200.7
Mercury, Total	EPA 245.1
	EPA 7470A
Selenium, Total	SM 18-19 3113B
Zinc, Total	EPA 200.7

Wastewater Metals III

Thallium, Total	SM 18-19 3113B

Wastewater Miscellaneous

Oil & Grease Total Recoverable	EPA 1664A

Sample Preparation Methods

EPA 3010A EPA 3510C EPA 5030B EPA 3020A

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All approved analytes are listed below:

Metals I	
Cadmium, Total	EPA 6010B
Chromium, Total	EPA 6010B
Lead, Total	EPA 6010B
Nickel, Total	EPA 6010B
Metals II	
Antimony, Total	EPA 6010B
Arsenic, Total	EPA 6010B
Mercury, Total	EPA 7471A
Selenium, Total	EPA 6010B
Nitroaromatics and Isophorone	
2,4-Dinitrotoluene	EPA 8270
2,6-Dinitrotoluene	EPA 8270
Isophorone	EPA 8270
Nitrobenzene	EPA 8270
Pyridine	EPA 8270
Nitrosoamines	
N-Nitrosodimethylamine	EPA 8270
N-Nitrosodiphenylamine	EPA 8270
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270
Phthalate Esters	
Bis(2-ethylhexyl) phthalate	EPA 8270
Diethyl phthalate	EPA 8270
Dimethyl phthalate	EPA 8270
Di-n-butyl phthalate	EPA 8270
Di-n-octyl phthalate	EPA 8270
Polychlorinated Biphenyls	
PCB-1016	EPA 8082
PCB-1221	EPA 8082
PCB-1232	EPA 8082
PCB-1242	EPA 8082
PCB-1248	EPA 8082
PCB-1254	EPA 8082
PCB-1260	EPA 8082
Polynuclear Aromatic Hydrocarbons	
Acenaphthene	EPA 8270
Anthracene	EPA 8270
Benzo(a)anthracene	EPA 8270
Benzo(a)pyrene	EPA 8270
Benzo(b)fluoranthene	EPA 8270
Benzo(ghi)perylene	EPA 8270
Chrysene	EPA 8270
Dibenzo(a,h)anthracene	EPA 8270

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All approved analytes are listed below:

Purgeable Halocarbons

Methylene chloride	EPA 8260B
Tetrachloroethene	EPA 8260B
·ans-1,3-Dichloropropene	EPA 8260B
,ichloroethene	EPA 8260B
Vinyl chloride	EPA 8260B

Purgeable Organics

2-Butanone (Methylethyl ketone)	EPA 8260B
Methyl tert-butyl ether	EPA 8021B

Sample Preparation Methods

EPA 1311 EPA 3050B EPA 3550B EPA 5035

·rial No.: 31689

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APPENDIX F
CERTIFICATE AND ANALYTE LIST FROM NH ELAP FOR ENDYNE, INC.
STATEMENT OF QUALIFICATIONS (ENDYNE, INC.)

The State of New Hampshire
Department of Environmental Services

Michael P. Nolin
Commissioner

October 18, 2006

Dr. Harry Locker
Endyne, Inc.
160 James Brown Drive
Williston, VT 05495

Subject: Renewed Accreditation.

Dear Dr. Locker:

Enclosed you will find your laboratory's renewed Certificate and Analyte List from the New Hampshire Environmental Laboratory Accreditation Program (NH ELAP). The Certificate and Analyte List shall be valid for the period stated provided you maintain a valid certificate from your Primary Accrediting Authority(s) or unless revoked or suspended for one or more reasons stated in rule Env-C 311.

The Analyte List is based upon the most recently received Analyte List from your Primary Accrediting Authority(s) and from the analytes available in NH ELAP's Field of Accreditation. Some Matrix – Technology / Method - Analyte combinations are not available, at this time, from NH ELAP. Review these documents closely.

Any change in your accreditation status from the Primary Accrediting Authority(s) must be forwarded to NH ELAP (i.e. an updated PAA Certificate / Analyte List). Specific changes to your analyte list, per accrediting authority, need to be noted in the cover letter or delays in future accreditation updates may be experienced.

Accreditation is required for any analyses that will be reported to NHDES WSEB.
Method accreditation does not imply acceptable for compliance testing.
Laboratory is required to use EPA approved methods where required by regulation.

Do not have copies of the laboratory's PT results forwarded to NH ELAP unless requested.

Contact me by e-mail if you have any questions at whall@des.state.nh.us. Program information is available at http://www.des.state.nh.us/nhelap/.

Sincerely,

Bill Hall
Program Manager

P.O. Box 95, 29 Hazen Drive, Concord, New Hampshire 03302-0095
Telephone: (603) 271-2513 • Fax: (603) 271-5171 • TDD Access: Relay NH 1-800-735-2964
DES Web site: **www.des.nh.gov**

PDF processed with CutePDF evaluation edition www.CutePDF.com

State of New Hampshire
Environmental Laboratory Accreditation Program

Awards Secondary Accreditation to

Endyne, Inc. (#2021)

of

Williston, VT

For the analyses listed on the attached page(s) in accordance with the provisions of the NELAC Standards and Env-C 300.

Certificate Number: 202106

Date of Issue: October 13, 2006

Expiration Date: October 12, 2007

Bill Hall
Program Manager

Method accreditation does not imply acceptable for compliance testing.
Laboratory is required to use EPA approved methods where required by regulation.
Continuing accreditation status is dependent on successful ongoing participation in the program.
Customers may verify the laboratory's current status by calling (603) 271-2998.

NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

Endyne, Inc. 160 James Brown Drive, Williston, VT 05495 (802) 879-4333
Analyte List Number: 202106-A Date of Issue: October 13, 2006 Expiration Date: October 12, 2007

GRANTS SECONDARY ACCREDITATION TO THE ABOVE MENTIONED LABORATORY FOR THE FOLLOWING ANALYSES:

DRINKING WATER MICROBIOLOGY

Coliform, Total	SM 18-20 9223

DRINKING WATER METALS

Antimony, Total	SM 18-19 3113B
Arsenic, Total	SM 18-19 3113B
Barium, Total	EPA 200.7
Cadmium, Total	EPA 200.7
Chromium, Total	EPA 200.7
Copper, Total	EPA 200.7
Iron, Total	EPA 200.7
Lead, Total	SM 18-19 3113B
Manganese, Total	EPA 200.7
Mercury, Total	EPA 245.1
Nickel, Total	EPA 200.7
Selenium, Total	SM 18-19 3113B
Silver, Total	EPA 200.7
Sodium, Total	EPA 200.7
Thallium, Total	EPA 200.9
Zinc, Total	EPA 200.7

DRINKING WATER INORGANIC CONTAMINANTS

Alkalinity	SM 18-20 2320B
Chloride	EPA 300.0
Color	SM 18-20 2120B
Corrosivity	SM 18-19 2330
Fluoride, Total	EPA 300.0
Hydrogen Ion (pH)	EPA 150.1
Nitrate (as N)	EPA 300.0
Nitrite (as N)	EPA 300.0
Orthophosphate (as P)	EPA 300.0
Solids, Total Dissolved	SM 18-20 2540C
Sulfate (as SO4)	EPA 300.0

MISC. DRINKING WATER ORGANICS

1,2-Dibromo-3-chloropropane	EPA 504.1
1,2-Dibromoethane	EPA 504.1

DRINKING WATER ACID, BASE / NEURALS

Bis(2-ethylhexyl) phthalate	EPA 525.2
Di (2-ethylhexyl) adipate	EPA 525.2

DRINKING WATER REGULATED INSECTICIDES

Aldrin	EPA 505
Chlordane Total	EPA 505
Dieldrin	EPA 505
Endrin	EPA 505
Heptachlor epoxide	EPA 505
Heptachlor	EPA 505
Lindane	EPA 505
Methoxychlor	EPA 505
Toxaphene	EPA 505

DRINKING WATER PCBs

PCB Screen	EPA 505

DRINKING WATER HERBICIDES

Dinoseb	EPA 515.2
Dicamba	EPA 515.2
Picloram	EPA 515.2
Pentachlorophenol	EPA 515.2
Dalapon	EPA 515.2
2,4,5-TP (Silvex)	EPA 515.2
2,4-D	EPA 515.2

DRINKING WATER TRIHALOMETHANES

Bromodichloromethane	EPA 524.2
Bromoform	EPA 524.2
Chloroform	EPA 524.2
Dibromochloromethane	EPA 524.2

This analyte list supersedes all previously issued analyte lists.

Bill Hall
Program Manager

Method accreditation does not imply acceptance for NHDES compliance testing.

Laboratory is required to use EPA approved / accepted methods where required by regulation.

Customers may verify the laboratory's current accreditation status by calling (603) 271-2998.

ACCREDITING nelap AUTHORITY

NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

Endyne, Inc. 160 James Brown Drive, Williston, VT 05495 (802) 879-4333
Analyte List Number: 202106-A Date of Issue: October 13, 2006 Expiration Date: October 12, 2007

GRANTS SECONDARY ACCREDITATION TO THE ABOVE MENTIONED LABORATORY FOR THE FOLLOWING ANALYSES:

DRINKING WATER VOLATILE ORGANICS

1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichlorobenzene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,3-Dichloropropane	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2,2-Dichloropropane	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chlorobenzene	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
Methylene chloride	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2

DRINKING WATER VOLATILE ORGANICS

tert-Butylbenzene	EPA 524.2
Tetrachloroethene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

DRINKING WATER INSECTICIDES (PESTICIDES)

Alachlor	EPA 525.2
Atrazine	EPA 525.2
Benzo(a)pyrene	EPA 525.2
Butachlor	EPA 525.2
Hexachlorobenzene	EPA 525.2
Hexachlorocyclopentadiene	EPA 525.2
Metolachlor	EPA 525.2
Metribuzin	EPA 525.2
Propachlor	EPA 525.2
Simazine	EPA 525.2

DRINKING WATER HALOACETIC ACIDS / ORGANIC DISINFECTION BY-PRODUCTS

Dibromoacetic acid	EPA 552.2
Dichloroacetic acid	EPA 552.2
Monobromoacetic acid	EPA 552.2
Monochloroacetic acid	EPA 552.2
Trichloroacetic acid	EPA 552.2

This analyte list supersedes all previously issued analyte lists.

Bill Hall
Program Manager

Method accreditation does not imply acceptance for NHDES compliance testing.

Laboratory is required to use EPA approved / accepted methods where required by regulation.

Customers may verify the laboratory's current accreditation status by calling (603) 271-2998.

ACCREDITING nelap AUTHORITY

NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

Endyne, Inc. 160 James Brown Drive, Williston, VT 05495 (802) 879-4333
Analyte List Number: 202106-A Date of Issue: October 13, 2006 Expiration Date: October 12, 2007

GRANTS SECONDARY ACCREDITATION TO THE ABOVE MENTIONED LABORATORY FOR THE FOLLOWING ANALYSES:

NON-POTABLE WATER MICROBIOLOGY

Coliform, fecal	SM 18-20 9222D

NON-POTABLE WATER METALS

Aluminum, Total	EPA 200.7
Antimony, Total	SM 18-19 3113B
Arsenic, Total	SM 18-19 3113B
Barium, Total	EPA 200.7
Beryllium, Total	EPA 200.7
Cadmium, Total	EPA 200.7
Chromium, Total	EPA 200.7
Copper, Total	EPA 200.7
Iron, Total	EPA 200.7
Lead, Total	SM 18-19 3113B
Manganese, Total	EPA 200.7
Mercury, Total	EPA 245.1
Nickel, Total	EPA 200.7
Selenium, Total	SM 18-19 3113B
Silver, Total	EPA 200.7
Thallium, Total	SM 18-19 3113B
Zinc, Total	EPA 200.7

NON-POTABLE WATER INORGANIC CONTAMINANTS

Ammonia (as N)	EPA 350.1
Biochemical Oxygen Demand	EPA 405.1
Chemical Oxygen Demand	EPA 410.4
Kjeldahl Nitrogen, Total	EPA 351.2
Nitrate (as N)	EPA 300.0
Nitrite (as N)	EPA 300.0
Oil & Grease Total Recoverable	EPA 1664A
Phosphorus, Total	EPA 365.1
Solids, Total Suspended	EPA 160.2
Solids, Total	EPA 160.3

PCBs IN NON-POTABLE WATER

PCB-1016	EPA 608
PCB-1221	EPA 608

PCBs IN NON-POTABLE WATER

PCB-1232	EPA 608
PCB-1242	EPA 608
PCB-1248	EPA 608
PCB-1254	EPA 608
PCB-1260	EPA 608

PESTICIDES IN NON-POTABLE WATER

4,4'-DDD	EPA 608
4,4'-DDE	EPA 608
Aldrin	EPA 608
alpha-BHC	EPA 608
Chlordane Total	EPA 608
delta-BHC	EPA 608
Dieldrin	EPA 608
Endosulfan I	EPA 608
Endosulfan sulfate	EPA 608
Endrin	EPA 608
Heptachlor epoxide	EPA 608
Heptachlor	EPA 608
Lindane	EPA 608
Methoxychlor	EPA 608
Toxaphene	EPA 608

VOLATILE ORGANICS IN NON-POTABLE WATER

1,1,1-Trichloroethane	EPA 624
1,1,2,2-Tetrachloroethane	EPA 624
1,1,2-Trichloroethane	EPA 624
1,1-Dichloroethane	EPA 624
1,1-Dichloroethene	EPA 624
1,2-Dichlorobenzene	EPA 624
1,2-Dichloroethane	EPA 624
1,2-Dichloropropane	EPA 624
1,3-Dichlorobenzene	EPA 624
1,4-Dichlorobenzene	EPA 624
2-Chloroethylvinyl ether	EPA 624
Benzene	EPA 624
Bromodichloromethane	EPA 624
Bromoform	EPA 624

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Bill Hall
Program Manager

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VOLATILE ORGANICS IN NON-POTABLE WATER

Bromomethane	EPA 624
Carbon tetrachloride	EPA 624
Chlorobenzene	EPA 624
Chloroethane	EPA 624
Chloroform	EPA 624
Chloromethane	EPA 624
cis-1,3-Dichloropropene	EPA 624
Dibromochloromethane	EPA 624
Dichlorodifluoromethane	EPA 624
Ethyl benzene	EPA 624
Methylene chloride	EPA 624
Tetrachloroethene	EPA 624
Toluene	EPA 624
Total Xylenes	EPA 624
trans-1,2-Dichloroethene	EPA 624
trans-1,3-Dichloropropene	EPA 624
Trichloroethene	EPA 624
Vinyl chloride	EPA 624

NON-POTABLE WATER SEMIVOLATILE ORGANICS

1,2,4-Trichlorobenzene	EPA 625
2,4,5-Trichlorophenol	EPA 625
2,4,6-Trichlorophenol	EPA 625
2,4-Dichlorophenol	EPA 625
2,4-Dimethylphenol	EPA 625
2,4-Dinitrophenol	EPA 625
2,4-Dinitrotoluene	EPA 625
2,6-Dinitrotoluene	EPA 625
2-Chloronaphthalene	EPA 625
2-Chlorophenol	EPA 625
2-Methyl-4,6-dinitrophenol	EPA 625
2-Nitrophenol	EPA 625
3,3' -Dichlorobenzidine	EPA 625
4-Bromophenylphenyl ether	EPA 625
4-Chlorophenylphenyl ether	EPA 625
4-Nitrophenol	EPA 625
Acenaphthene	EPA 625
Acenaphthylene	EPA 625
Anthracene	EPA 625
Benzidine	EPA 625

NON-POTABLE WATER SEMIVOLATILE ORGANICS

Benzo(a)anthracene	EPA 625
Benzo(a)pyrene	EPA 625
Benzo(b)fluoranthene	EPA 625
Benzo(ghi)perylene	EPA 625
Benzo(k)fluoranthene	EPA 625
Benzyl butyl phthalate	EPA 625
Bis (2-chloroisopropyl) ether	EPA 625
Bis(2-chloroethoxy)methane	EPA 625
Bis(2-chloroethyl)ether	EPA 625
Bis(2-ethylhexyl) phthalate	EPA 625
Chrysene	EPA 625
Dibenzo(a,h)anthracene	EPA 625
Diethyl phthalate	EPA 625
Dimethyl phthalate	EPA 625
Di-n-butyl phthalate	EPA 625
Di-n-octyl phthalate	EPA 625
Fluoranthene	EPA 625
Fluorene	EPA 625
Hexachlorobenzene	EPA 625
Hexachlorobutadiene	EPA 625
Hexachlorocyclopentadiene	EPA 625
Hexachloroethane	EPA 625
Indeno(1,2,3-cd)pyrene	EPA 625
Isophorone	EPA 625
Naphthalene	EPA 625
Nitrobenzene	EPA 625
N-Nitrosodimethylamine	EPA 625
N-Nitrosodi-n-propylamine	EPA 625
N-Nitrosodiphenylamine	EPA 625
Pentachlorophenol	EPA 625
Phenanthrene	EPA 625
Phenol	EPA 625
Pyrene	EPA 625

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ACCREDITING nelap AUTHORITY

Statement Of Qualifications

Introduction

Endyne Inc., located in Williston, VT is a full-service Environmental Testing Laboratory specializing in organic, inorganic, metal and microbiological analyses. Endyne has extensive experience testing a variety of samples including: water, wastewater, soils hazardous waste and air. Endyne's mission is to provide the environmental community with high quality analytical data in a timely and cost effective manner.

Certifications

- ELAP Certification
- New Hampshire Certification

Experience

Endyne has a broad and diverse range of project experience ranging from the analysis of drinking water to monitoring large scale subsurface remediation projects. Examples of the types of projects in which Endyne has participated include: Hazardous waste characterization, Industrial and Municipal discharge compliance, Phase II/V Federal drinking water compliance, Landfill monitoring, Industrial hygiene testing, Underground storage tank testing, Sludge Land application studies, Dairy waste testing, TCLP/Priority Pollutant analyses and much more.

Quality Control/Quality Assurance

Each analysis is of paramount importance to the Laboratory; and, as such, each analysis is subject to a rigorous QA/QC procedure. The Laboratory's industry standard QA/QC plan includes analysis of QC samples, spikes, duplicates and blanks to insure data quality.

The Laboratory maintains a variety of certifications in Vermont, New Hampshire, and New York. In accordance with certification requirements, the laboratory participates biannually in several EPA sponsored blind performance evaluation studies. Further, the Laboratory is routinely visited by Certification Officers to review laboratory procedures and operations first hand.

Capabilities

Endyne is truly a full-service Laboratory and maintains a staff of 18 professionals in a modern 6000 square foot facility. The Laboratory is equipped with state of the art instrumentation in each of the three divisions of the Laboratory. The Organics Laboratory is equipped with several Hewlett-Packard GC, GC/MS and HPLC systems for the analysis of environmentally significant organic compounds. Available GC detectors include: MS, ECD, FID and PID. The Laboratory routinely processes samples for EPA 8000 and 600 series including: 8020, 8100, 8260, 8270, 601, 602, 608, 610 and 625. The Laboratory also operates a field portable GC for remote field operations.

The Metals Analysis Laboratory is equipped with a Graphite Furnace Atomic Absorption Spectrophotometer (GFAA) and a Leeman Labs Sequential Inductively Coupled Plasma Atomic Emission Spectrophotometer (ICP) for the analysis of metal species. The Laboratory processes samples in accordance with EPA methods 6010 and 200.7 for the ICP, and SM 3113B and 200 Series for the GFAA.

The Inorganic Laboratory, or wet lab, is equipped with all the instrumentation necessary to perform gravimetric, titrimetric and spectrophotometric assays. The Laboratory has been performing all the testing required under the NPDES discharge requirements since 1987.

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Laboratory QA/QC Policy Statement

Each individual analysis that is performed by Endyne is of critical importance to the laboratory. It is thus imperative that Endyne be able to assure the validity of each result that is reported. To this end, Endyne observes the QA\QC protocol detailed below. In summary, the strategy consists of:

1. Participation in NELAP approved Performance Evaluation Tests for both drinking water and waste water.

2. Calibration verification standards analyzed daily.

3. Analysis of independent QC standards every 10 samples and continued charting of this data to define method precision and accuracy.

4. Monthly performance of intra-laboratory performance tests.

5. Duplicate laboratory analyses every 20 samples.

6. Analysis of matrix spike samples every 20 samples.

7. Analysis of laboratory blanks every 20 samples.

8. For appropriate methods, surrogate analytes are evaluated to monitor the performance of each individual analysis.

9. Review of calculations from data and checking of reported values.

APPENDIX G

STANDARD OPERATING PROCEDURES (SOPS) BY ENDYNE, INC.:

VOLATILE ORGANIC COMPOUNDS METHOD EPA 8260/8260B SOP
SEMI-VOLATILE ORGANIC CONTAMINANTS METHOD EPA 8270C SOP
ORGANOCHLORINE PESTICIDES METHOD EPA 8081A
POLYCHLORINATED BIPHENYLS (PCBS) METHOD EPA 8082
ICP SOP EPA METHOD 6010B
MERCURY IN SOLID OR SEMISOLID WASTE METHOD SW 7471A SOP
TOTAL ORGANIC CARBON – LABORATORY QC LIMITS
SOP: TOC IN SEDIMENT
SOP: TOC IN WATER

Endyne, Inc.

160 James Brown Dr.
Williston, VT 05495
Phone: (802) 879-4333
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VOLATILE ORGANIC COMPOUNDS
Method EPA 8260/8260B
Standard Operating Procedure

REVISED: April 7, 2005

Revision #: 8

Technical Director: _____Date : _____
Harry B. Locker Ph.D.

QA Officer: _____Date : _____
Mark L. Fausel

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I. Overview:

-Method 8260 is used to determine volatile organic compounds (VOCs) in a variety of waste matrices, including waters, soils, sludges, waste solvents and petroleum wastes. Samples are purged with an inert gas (N_2). Analytes are captured on a carbon packed sorbent trap, then back flushed with a stream of Helium into a capillary GC Column interfaced to a mass spectrometer.

-The following compounds are determined by this method:

Dichlorodifluoromethane
Chloromethane
Vinyl Chloride
Bromomethane
Chloroethane
Trichlorofluoromethane
Diethyl Ether
Acetone
1,1-Dichloroethene
Methylene Chloride
Carbon Disulfide
Methyl-t-butylether (MTBE)
trans-1,2-Dichloroethene
1,1-Dichloroethane
2-Butanone
2,2-Dichloropropane
cis-1,2-Dichloroethene
Chloroform
Bromochloromethane
Tetrahydrofuran (THF)
1,1,1-Trichloroethane
1,1-Dichloropropene
Carbon Tetrachloride
1,2-Dichloroethane
Benzene
Trichloroethene
1,2-Dichloropropane
Dichlorobromomethane
Dibromomethane
4-Methyl-2-pentanone
cis-1,3-Dichloropropene
Toluene
trans-1,3-Dichloropropene
1,1,2-Trichloroethane
Isopropyl Alcohol
ETBE
TAME
Acrylonitrile
2-Hexanone
1,3-Dichloropropane
Tetrachloroethene
Dibromochloromethane
1,2-Dibromoethane
Chlorobenzene
1,1,1,2-Tetrachloroethane
Ethylbenzene
m/p-Xylene
o-Xylene
Styrene
Bromoform
Isopropylbenzene
1,1,2,2-Tetrachloroethane
1,2,3-Trichloropropane
n-Propylbenzene
Bromobenzene
1,3,5-Trimethylbenzene
2-Chlorotoluene
4-Chlorotoluene
tert-Butylbenzene
1,2,4-Trimethylbenzene
sec-Butylbenzene
p-Isopropyltoluene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
n-Butylbenzene
1,2-Dichlorobenzene
1,2-Dibromo-3-chloropropane
1,2,4-Trichlorobenzene
Hexachlorobutadiene
Naphthalene
1,2,3-Trichlorobenzene
Methyl isothiocyanate (MITC)
Isopropyl ether
t-Butanol
Acrolein

II. Safety:

This method involves the handling of the chemicals outlined below. Each of these chemicals may have specific associated hazards. Analysts must read and become familiar with all elements of each chemical's MSDS. Make special awareness of the symptoms of exposure and potential routes of entry. Analysts must notify the Safety Officer immediately if any symptoms of exposure are suspected.

PPE must be worn at all times in designated work areas. All applicable engineering controls, such as fume hoods or splash shields, must be used when performing analyses. To ensure the safety of all laboratory personnel; work areas must be kept neat and organized, equipment and bench-tops must be cleaned and sterilized, if necessary, at the completion of each task, all containers must be properly labeled as to contents and hazard ratings and spills cleaned immediately and completely.

A. Lab coats, Nitrile Gloves and Protective Eye Glasses are to be worn at all times during analysis by this method and when handling any laboratory sample.
B. A thorough MSDS review of Hydrochloric Acid, Methanol, Ascorbic Acid, Sodium Thiosulfate and all laboratory calibration standards should be performed prior to analysis by this method.
C. All standards should be made in a fume hood away from heat sources.
D. Several analytes tested by this method have been classified as human carcinogens. Stock standards of these materials should be handled in fume hoods. Toxic gas respirators should be won when handling concentrated standards and samples.

III. Waste Disposal:

A. Expired standards, QCs and misc. solvents are disposed of in the flammable liquids drum in the hazardous waste room.
B. Non-contaminated ("non-hot") samples can be dumped down the drain with a stream of running water.
C. Contaminated ("Hot") samples are disposed of in the corrosive liquids drum in the hazardous waste room.
D. Soil samples are disposed of in the soil waste drum in the hazardous waste room.

IV. Sample Collection, Preservation and Storage:

A. Sample holding time is 14 days from the sample time.
B. All liquid samples should be sampled in duplicate, pH adjusted to < 2 with HCl and stored at 4°C with no headspace.
C. All soil samples should be sampled in duplicate and stored at 4°C with no headspace.
D. Refer to Appendix A for MeOH preservation sample collection (if samples are methanol preserved in the field).
E. Air Samples collected in tedlar bags should be analyzed within 8 hours of sampling

V. Equipment and Supplies:

-GC: Hewlett Packard 5890 with Jet Separator
-MS: Hewlett Packard 5970 Series
-GC: Hewlett Packard 5890 Split/Splitless option
-MS: Hewlett Packard 5971 Series
-Miscellaneous Syringes & Vials
-Analytical Balance
-OI Analytical 4560 sample concentrator with MPM 16
-OI Analytical 4560 sample concentrator with DMP 16

VI. Reagents:

-Reagent Grade DI Water
-Methanol (Purge & Trap Grade or Better)
-Volatile Mix Standard (NSI Q2479 Custom Volatiles Mix - 200 ppm)
-Internal Standard Mix (AccuStandard STM - 341N 2000 ppm
-Surrogate Standard Mix (AccuStandard STM 330N 2000 ppm)
-Hydrochloric Acid
-2-Chloroethylvinylether Standard (AccuStandard M-601C-10X 2000ppm)
-Oxygenated Gas Additive Standard (AccuStandard OGAD-001 2000ppm)
-Priority Pollutant Standards (Acrylonitrile APP-9-008; Acrolein APP-9-007 100ppm)
-Mics neat standards (Isopropanol – Fisher Sci ; MITC – Sigma Aldrich)

VII. Interferences:

A. Special precaution must by taken to isolate samples from all atmosphere Methylene Chloride contamination. $MeCl_2$ will permeate through PTFE tubing. It is readily carried on clothing exposed to fumes during common liquid/liquid extractions creating random background levels.
B. Interfering contamination may occur when a sample containing low concentration of VOCs is analyzed immediately after samples containing high concentrations of VOCs. Late eluting analytes tend to “carry over” in greater abundance than early eluting analytes.
C. Trace levels of Ketones, Methylene Chloride, Chloroform and other organic solvents can be present in high purity methanol. Analysis of each new lot of methanol should be performed prior to use.
D. Impurities from purge and carrier gas can be minimized with gas traps. Trihalomethanes may occur in blanks as traps from the DI water system age. Replace traps as needed.

VIII. Quality Control:

A. Initial demonstration of capability (IDC) is performed by analysts training under the supervision of analysts familiar with interpreting GC/MS data and updated annually thereafter. Current IDCs and continuing Demonstration of Capability (DOC) studies are stored in analyst personnel files and are also filed with the VOC QA/QC data. Electronic copies of current DOCs are located on “Wayne” (C:EXCELQC/QC/IDC).
   1. Four replicate aliquots of mid-calibration reference standards are analyzed by the procedure outlines in section IX

2. Calculate the average recovery, the standard deviation and the % standard deviation of the recoveries for each analyte of intrest.
3. Results of IDC are compared to laboratory control charts to see if acceptance criteria are met. In the absence of laboratory acceptance criteria recoveries of 80-120% can be used as guidance in evaluating the results.
4. If any % standard deviation fails precision limits of 20% or any mean fails accuracy limits (+/-20% or control limit value, whichever is lower), the source of the problem must be found and corrected. Analytes that failed must be reanalyzed and acceptance criteria must be met.

B. Method detection limits (MDL) study is performed annually to determine acceptable detection limits. Current MDL studies are stored with the VOC QA/QC data. Electronic copies of current MDLs are located on "Wayne" (C:EXCELQC/QC/MDL).
   1. Seven reference standards at 2.5 times the minimum reporting limit are analyzed according to the procedure outlines in section IX
   2. The standard deviation of the seven most precise values for each analyte are multiplied by 3.14 to determine the MDL value.
   3. The calculated MDL value must be 2.5 – 5 times less than the target of the reference standards or the MDL is reanalyzed.
   4. The calculated MDL value must be lower than reporting limits outlined in Tbl.2

C. Method Reporting Limit determination
   1. Laboratory reporting limit is either the concentration of the lowest analyzed calibration standard or the calculated MDL value for that analyte, whichever value is higher.
   2. Reporting limits can be increased for analytes with poor purge efficiencies, analytes present in the laboratory atmosphere or analytes which tend to carry over from one run to the next as long as the increased reporting limits are lower than minimum requirements for the laboratory regulating bodies (NELAC and State of Vermont).

D. 4-Bromofluorobenzene analysis (BFB), a direct injection of BFB is made at the beginning of each data set and ever 12 hours of sample analytical time. The mass intensities are then compared to the tune criteria listed in Table 1.
   1. If all tune criteria is not met, system maintenance and reanalysis of the BFB are required until performance is acceptable.
   2. A maximum of 50ng of BFB is analyzed at the start of each data set.
   3. Continuous monitoring of BFB tune criteria may be observed with each sample analyzed as BFB is use as a method surrogate.

E. Laboratory control standards; a blank, an independent fortified blank and a line check standard are performed with each data set.
   1. Analytes from laboratory reagent blanks should be 10 times less than the highest reported value for the data set and below method detection limits to confirm the instrument and laboratory are reasonably free of contamination. System maintenance, removal of laboratory contaminants and reanalysis of blank are required if blank criteria are not met. All data is qualified if blank criteria are not met.

2. Analytes from field and trip blanks should be below method detection limit to confirm sampling techniques are free of contamination.
3. Independent fortified standards and line check standards are tabulated prior to sample analysis. Percent recoveries should fall within acceptable limits tabulated in the method control charts or should be from 70-130% of the true value, whichever levels are more stringent. System maintenance and reanalysis of standards is required prior to sample analysis. All data is qualified if recoveries are outside tabulated acceptance levels.
4. Control charts are tabulated annually from the percent recoveries of the continuous line check and independent fortified standards. Upper and lower warning limits are ± 2 standard deviations of the mean. Upper and lower control limits are ± 3 standard deviations of the mean. Control limits are posted at the VOC work station and are stored on "Wayne" at C:EXCELQC/QC/CONTROL.
5. A graphic print out of each analytes recovery is performed annually so recovery trends can be easily viewed.

F. Calibration line quality control
1. The relative standard deviation (RSD) of the initial calibration should be less than 15% for each analyte. Reanalysis of calibration standards and acceptable RSD levels must attained prior to sample analysis.

G. Instrument and software performance
1. Good column and GC performance should produce symmetrical peaks.
2. Software should recognize and integrate 99% of compounds analyzed or instrument maintenance is required.
3. Entire symmetrical peak for each analyte must elute in the retention time window establish in the initial calibration. Instrument maintenance is require if analytes fall outside retention time windows.

H. System performance check compounds (SPCC) are performed daily from the line check standard. Minimum acceptable average RFs should be > 0.100 for chloromethane, 1,1-dichloroethane and Bromoform. Minimum acceptable average RF should be > 0.300 for Chlorobenzene and 1,1,1,2-Tetrachloroethane. See calibration and standardization section for RF calculation.

I. Calibration check compounds (CCC) are performed daily from the line check standard. The RF % difference for 1,1 Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene and Vinylchloride should be < 25% of the initial calibration.

$$\% \text{ Difference} = \frac{(\text{RF of Initial Cal} - \text{RF of Current Check std})}{\text{RF of Initial Cal}} \times 100$$

J. Matrix spike and matrix spike duplicate (MSD) samples are analyzed every 20 samples or with each data set. Percent recoveries are tabulated with the LFB data and are subject to the same acceptance criteria previously stated. Analyte percent difference from the MSD must be less than 20% or system maintenance and reanalysis are required. If it is determined MSD data is outside laboratory control limits due to sample matrix interferences, reported data will be qualified.

IX. Procedure:

A. Instrument Operating Conditions
1. 5970 gc/ms set up
   a. Trap: VOCARB 4000 Supelco
   b. Column: 60m; 0.53mm ID; 3.0um dF Restek (RTX-624)
   a. Carrier Flow (He); 20mls/min, 10mls/min make up gas
   d. Purge Flow (N2); 20mls/min
   e. Source vacuum; 6 x 10-6 torr (approximate)
   f. Purge time 11 min; desorb time 4 min; bake time 20 min
   g. Acquisition parameters saved in method 8260aq.m are as follows:
      -Sample Inlet: GC
      -Injection Source: External Device
      -Injection Location: Front
      -Temperature Zones for Inlet B, Detector A, and Detector B are all set at 250°C.
      -Oven Program Initial Temp: 40°C
      -Oven Program Initial Time: 1min
      -Temperature Gradient Level 1: Rate 5.5°C/min; Temp 70°C; Time 1min
      -Temperature Gradient Level 2: Rate 6.5°C/min; Temp 210°C; Time 3min
      -Total Run Time: 31.99min
2. 5971 gc/ms set up
   a. Trap: VOCARB 4000 Supelco
   b. Column: 60m; 0.32mm ID; 1.8um dF Restek (RTX-624)
   c. Constant flow inlet B ON 9.0 PSI (He)
   d. Purge Flow (N2); 20mls/min
   e. Source vacuum; 5.3 x 10-5 torr (approximate)
   f. Purge time 11 min; desorb time 4 min; bake time 20 min
   g. Acquisition parameters save in method aq-8260.m are as follows:
      -Sample Inlet: B
      -Pressure 8.94
      -Flow 1.5
      -Velocity 31.4
      -Split 4.5
      -Ratio 3:1
      -Initial Set points – valves 3and 4 are on , all others valves and relays off
      -Initial Temp. 50C; ms on at 3.0 min
      -Gradient is 5.5 c/min to final temp of 210 – hold final time 1.73min

B. BFB direct injection: performed at the beginning of each analytical set and every 12 hours of analysis on both instruments
1. Activate the MS Top Software
2. Select “Sequent-Edit sample log table”
3. Enter data file: date/year/cycle#
4. Enter method: “BFBA” for 5970 and “AQ-BFB” For 5971
5. Enter sample information: “50ng BFB” and press “ok”
6. Select “sequence-run”, enter “full method”
7. Data file directory “c:\hpchem\1\data\feb03”, press “ok”
8. When temperatures on GC oven equilibrate a “start” message will appear on the acquisition computer. Inject 2µL of 25ppm 8260 IS/SURR mix.

9. Press start on GC oven
10. Confirm BFB spectral scan and ion abundance criteria are calculated, plotted and printed. Reanalysis of BFB and instrument maintenance must be performed if any abundance criteria is not met.

C. Sequence Set Up
1. Enter and save sample information in GC/MS daily run log stored at "C:\msoffice\excel\rpt\elvsdail.xls" on the 5970 system and "C:excelfiles\runlog02". Include date, tube number, file name, reference number, reference number suffix, sample preservation, client, project, site dilutions and miscellaneous notes. For soil analysis include injection volume, sample mass and extraction volume.
2. Load samples at appropriate dilutions on MPM 16 and DPM 16
3. Press "SPL" on sample concentrator and enter range of tubes to be analyzed. Press "start".
4. When sample concentrator indicates "DSB-RDY" activate ms top software
5. Program sequence-enter data file names to be analyzed, aquisition method is "8260Aq"on 5970 and "aq-8260" on 5971 work station.
6. At sample info section enter laboratory reference number, dilution and miscellaneous sample information then press "ok"
7. Select "sequence-run", enter "full method"
8. Data file directory "c:\hpchem/1/data/mmmyy", press "ok", sequence will start automatically when temperatures equilibrate.

D. Sample preparation and Processing
1. Add 10uL IS/Surr mix to each 5 mls of sample, QC, Line Check Blank and spike prior to loading purge and trap.
2. Run file through current method in MS ChemStation software after all quality control criteria have been met. Check the integration of each peak to be quantitated upon.
3. Check surrogate recoveries to be certain they are within established control limits.
4. Soil/Sludge in house extraction
   a. Weigh approximately 10g of soil on an analytical balance into a 40ml vial
   b. Add 5ml purge and trap grade methanol.
   c. Shake samples vigorously for 2min and allow samples to settle refridgerated for 24 hours
5. Tedlar bags are analyzed at first available opportunity.
   a. With gas tight syringe, extract no more than 100cc sample
   b. When autosampler cycles to purge on a clean tube, open sample valve and introduce sample at approximately 20mls /min.
   c. Process results and associated QA/QC via external standard method of most recent calibration.

X. Calibrations and Standardization (make/use/storage):

A. Prepare a 10 ppm Working Standard in methanol containing all 8260 analytes:
-50µL 200 ppm NSIQ2479 Custom VOC mix
-5µL 2000 ppm Accustandard M-601C-10x
-945µL Purge and Trap Methanol
-Store at < 0C in organic standard freezer

B. Prepare a 25 ppm Internal Standard/Surrogate Solution:
-12.5µL 2000 ppm STM-341N IS Mix
-12.5µL 2000 ppm STM-330N Surr Mix
-975µL Purge and Trap MeOH.
-Store at < 0C in organic standard freezer

C. Analyze a series of calibration standards at the following concentrations 1.0 ppb, 2.0 ppb, 5.0 ppb, 10 ppb, 20 ppb, 50 ppb, 100 ppb, 150ppb and 200 ppb following the steps outlines in section IX.

1. Good GC and column performance will produce symmetrical peaks with minimal tailing.
2. The GC/MS software should recognize a GC peak in the appropriate retention time window. If fewer than 99% of the compounds are recognized, system maintenance is required.
3. Calculate the response factor for each analyte, surrogate and isomer pair for each analyte.

$$RF = \frac{A_x \; Q_{is}}{A_{is} \; Q_x}$$

A_x = Quantitation Ion Abundance of the Analyte
A_{is} = Quantitation Ion Abundance of the Internal Standard
Q_x = Quantity of Analyte Purged
Q_{is} = Quantity of Internal Standard

4. Calculate the mean RF from analysis of calibration standards.
5. Calculate the standard deviation and relative standard deviation from each mean: RSD = 100 (SD/M). If the RSD exceeds 15% reanalyze additional aliquots of cal solution to obtain acceptable RSDs.

D. Evaluation of Retention Times

1. The relative retention time of each target analyte should agree within 0.06 RRT units

E. Peak Integration

1. Peak integration should be consistent for all analytes, and especially for the integration of standards and compound of a sample.
2. Single peaks, the baseline should begin at the start of the upward slope of the peak and end at the bottom of the downward slope.

3. If there is a raised baseline, the analyst should be careful to not integrate the raised baseline as part of the area of the target peak. Co-

elution of peaks, the baseline should start at the beginning of the upward slope of the peak and remain level until the slope of the valley of the two peaks is at its lowest level. If the target peak is the second of a co-eluting pair, the baseline should be drawn backwards to the same level.

XI. Data Reduction and Interpretation:

A. Calculate analyte and surrogate concentrations in water samples:

Cx = [(Ax) (Qis) 1000 ÷ (Ais) (RF) (V)]
V = Volume water in mls
RF = Response factor from initial calibration
C_x = Concentration in µg/L
A_x = Integrated Abundance of the quantitation ion of the Analyte
A_{is} = Integrated Abundance of the quantitation ion of the Internal Standard
Q_{is} = Quantity of Internal Standard added to the sample

B. Analyte concentration in soil samples:

$$Cs = C_x \times \frac{5}{M \times P} \times \frac{V_E + ((M*(1-P))*1000)}{V_I}$$

Where:
C_s = analyte concentration as µg/kg, dry
C_x = analyte concentration from instrument expressed as ng/ml
M = sample mass in grams
P = percent solid expressed as a decimal extraction
V_E = extraction volume in µL
V_I = Injection volume in µL

C. Qualitative Analysis-Compound identification is determined by satisfying two criteria.
1. Elution of sample compound at the same GC relative retention time as the standard analyte ± 0.06 RRT units.
2. Comparison of sample and standard mass spectrum.
   a. All ions greater than 10% of the primary ion in the standard must be present in the sample spectrum.
   b. Relative intensities of secondary ions must agree within + or - 20%.
   c. Identification of compounds not in the calibrated standard is determined by comparing to library mass spectra

XII. Data Recording:

A. Sample data is electronically imported to the LIMS from the VOC/GCMS computer after it has been reduced to a usable format. Current data reduction MACRO is LIMSMAC.XLM stored at c:\EXCELFILES\REPORTS. on the VOC/GCMS computer.
B. Record any Instrument Maintenance in the maintenance log for the PT/GC/MS
C. All standards opened and made are reported in the VOC standards log
D. Individual calibration standard and associated QC dilutions are recorded in the daily run log.
E. Sample pH is measured using pH strips and recorded on the sample chain of custody and in the daily run log.
F. Analyst initials are written on each sample chromatogram. Sample chromatograms are filed sequentially with other organic chromatograms and stored for a minimum of 3 years.
G. All QC, Line Check, Blank data and BFB Tune Criteria is printed and stored sequentially by date with the VOC/GCMS QC Data and is stored for a minimum of 5 years.
H. Individual QC and Line Check data is entered to 8260 QC Spread Sheets to determine if acceptable QA/QC Criteria has been attained prior to reporting each analytical batch. Data is stored on VOC/GCMS computer (c:\EXCELQC\QC\CONTROL) and is tabulated and printed annually.
I. Sample data is entered to LIMS with Analyst ID, Date, Units and analyte concentration.
J. A hard copy of all quality control data is stored with the PT/GCMS data
K. A hard copy of the instrument run log is printed monthly and stored with the QC data by the GC/MS work station.
L. Computer hard drive is backed up quarterly to a CD. This includes run log, QC charts, sample data, line standards, methods and QC standards.
M. Method hard copies are printed and stored with the PT/GCMS QC hard copies.

XIII. Instrument Maintenance/Trouble Shooting:

A. Injection port septa is replaced every 20 injections.
B. Poor response or failed tune criteria may be indicative of a dirty MS source. Follow procedures in the HP MSD 5970 operators manual to clean the source.
C. No sample response indicates a worn of broken filament. Follow procedures in the HP MSD 5970 operators manual to replace filaments or manually switch to alternate filament on back of MSD.
D. A high baseline or poor instrument response may indicate a leak in the system. Leaks will oxidize the column and shorten column life. Perform an air and water check following any instrumental maintenance.
E. Varying surrogate and analyte response and excessive analyte carry over are common with deteriorating traps. Replace the Vocarb 4000 in the Tekmar LCS-2 according to the operating manual.
F. Varying response may indicate a contaminated injection port. Replace the injector liner or Salanize an old injection liner in accordance to HP GC oven 5890 operators manual.

XIV. Method Modifications:

XV. Bibliography/References:

EnviroQuant Chemstation, G1701BA Version b.01-00 © Hewlett-Packard 1989-1998

HP 5971A Mass Selective Detector Hardware Manual, edition 1 © Hewlett-Packard Feb. 1990, Publication # 05971-90011

HP 5890A Series II Programmable Cool on Column Inlet, eighth edition © Hewlett-Packard, Sept. 1995 Publication # 05890-90300

HP 5890A Series II Reference Manual, fifth edition © Hewlett-Packard, Sept. 1991Publication # 05890-90270

Method 8000b Determinative Chromatographic Separations, EPA SW-846 NTIS ,CD ROM Version 2, Dec. 1997

Method 8260b– Volatile Organic Compounds by Gad Chromatography/Mass Spectrometry (GC/MS), Revision 2, US EPA December 1996

XVI. Revision History:

Revision #7 January 26, 2004

- -Added Bibliography/Reference section; Added Revision History section; Added sign off sheet.
- -Edited typos; formatted tab sets and spacing; updated table of contents
- -Added laboratory safety requirements.
- -Added NH gas additive compounds; Added priority pollutant compounds; Added OMYA compounds.
- -Added Tedlar bag requirements.

Revision #8 – April 7, 2005

- -Edit for content and grammer.

TABLE 1

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50	15 to 40 % of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Appendix A

VOLATILE ORGANIC COMPOUND SAMPLING WITH METHANOL PRESERVATION

In light of recent research conducted on analyte recovery efficiency, Endyne is recommending to its clients that they modify their sampling procedure for Volatile Organic Compounds (VOCs) in soil/solid matrices; SW-846 Method 5035.

The recommended protocol calls for the sampler to notify the laboratory approximately 1 week before sampling date. Once notified the laboratory will prepare 40-ml vials for each site.

Preparation will involve adding exactly 10.0 ml of a high purity grade methanol (MeOH) to a 40-ml vial and determining the exact weight, which will be recorded on the vial along with the date prepared. A pen mark is made on the label to note the meniscus of the solvent at the time of preparation.

The sampler will then add approximately 20.0 g of soil in the field to each vial. This is roughly equivalent to 2 rounded tablespoons or 1.5 inches in the vial of a loosely packed soil or one heaping tablespoon of a denser/damper soil. **Once the soil has settled, there should be approximately one centimeter of MeOH as a supernatant.** The sampler should mark on the bottle the level of the meniscus after sample addition, this will allow the laboratory to confirm that there was no sample loss at the time of analysis.

The sampler will also include a 40.0-ml vial of soil sampled the standard way. This vial will be used to determine percent solids as well as a back up in case of sample loss with the MeOH vial.

Once returned to the lab, the soil is ready for analysis. The weight of the sample will be determined by the difference of the before and after weights.

Advantages:

-More accurate and generally higher analyte recoveries.

Disadvantages:

-Poor sampling/handling procedure can result in greater erroneous results.

Hints:

- It is very important to not splash any of the MeOH out of the vial when collecting the sample.
- Do not add any additional labels to the vial, as it will affect the sample weight.
- Keep vials refrigerated/even before sampling.
- Do not leave caps open for very long since MeOH evaporates quite quickly.
- After sampling, be sure to wipe the threads of the vial & cap with a clean tissue. Even the slightest amount of dirt can prevent a good seal.
- Unused sample vials are stable for several weeks when refrigerated. Always confirm that no methanol has evaporated from the vial by checking the meniscus line.

Table 2
TABLE 2-SW 8260B Water Reporting Limits

Parameter	Limit ug/L	Parameter	Limit ug/L
Acetone	10.0	1,1-Dichloropropene	1.0
Benzene	1.0	cis-1,3-Dichloropropene	1.0
Bromobenzene	1.0	trans-1,3-Dichloropropene	1.0
Bromochloromethane	2.0	Diethyl Ether	10.0
Bromodichloromethane	1.0	Ethylbenzene	1.0
Bromoform	1.0	Hexachlorobutadiene	5.0
Bromomethane	5.0	2-Hexanone	10.0
2-Butanone	10.0	Isopropylbenzene	1.0
n-Butylbenzene	1.0	p-Isopropyltoluene	1.0
sec-Butylbenzene	1.0	Methylene Chloride	10.0
tert-Butylbenzene	1.0	4-Methyl-2-Pentanone	10.0
Carbon Disulfide	10.0	MTBE	2.0
Carbon Tetrachloride	1.0	Naphthalene	2.0
Chlorobenzene	1.0	n-Propylbenzene	1.0
Chloroethane	5.0	Styrene	1.0
2-Chloroethyl Vinyl Ether	20.0	1,1,1,2-Tetrachloroethane	2.0
Chloroform	1.0	1,1,2,2-Tetrachloroethane	2.0
Chloromethane	3.0	Tetrachloroethene	1.0
2-Chlorotoluene	1.0	Tetrahydrofuran	10.0
4-Chlorotoluene	1.0	Toluene	1.0
Dibromochloromethane	1.0	1,2,3-Trichlorobenzene	2.0
1,2-Dibromo-3-Chloropropane	2.0	1,2,4-Trichlorobenzene	2.0
1,2-Dibromoethane	2.0	1,1,1-Trichloroethane	1.0
Dibromomethane	2.0	1,1,2-Trichloroethane	1.0
1,2-Dichlorobenzene	1.0	Trichloroethene	1.0
1,3-Dichlorobenzene	1.0	Trichlorofluoromethane	2.0
1,4-Dichlorobenzene	1.0	1,2,3-Trichloropropane	2.0
Dichlorodifluoromethane	5.0	1,2,4-Trimethylbenzene	1.0
1,1-Dichloroethane	1.0	1,3,5-Trimethylbenzene	1.0
1,2-Dichloroethane	1.0	Vinyl Chloride	2.0
1,1-Dichloroethene	1.0	Xylenes, Total	2.0
cis-1,2-Dichloroethene	1.0	Isopropanol	50.0
trans-1,2-Dichloroethene	1.0	MITC	5.0
1,2-Dichloropropane	1.0	ETBE	2.0
1,3-Dichloropropane	1.0	TAME	2.0
2,2-Dichloropropane	1.0	t-Butanol	10.0
Isopropyl ether	2.0	Acrylonitrile	20.0
Acrolein	20.0		

Note: Soil reporting limits are base on the sample percent solid, extraction mass, and extraction solvent volume and therefore vary with each individual sample. See section XI for soil calculation conversion to ug/kg, dry.

Note: Current MDL study can be referenced at “Wayne” C:/EXCELQC/QC/MDL. Hard copied and raw data are filed with VOC QC data.

I acknowledge that I have read this Standard Operating Procedure and agree to adhere to the Safety and Quality Control policies outlined within.

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SEMI-VOLATILE ORGANIC CONTAMINATS
METHOD EPA 8270C

Standard Operating Procedure

REVISED: April 6, 2005

Revision #: 7

Technical Director: _____Date :_____
Harry B. Locker Ph.D.

QA Officer: _____Date :_____
Mark L. Fausel

Document Control Copy on Buff colored paper. Any other color is not official version

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I. Overview:

A. Method 8270 is used to determine the concentration of semi-volatile organic compounds in extracts prepared from all types of waste matrices, soils, and groundwater.

B. Target Analytes

Base/Neutral Extractable:

Anthracene	Azobenzene	Benzidine
Benzo(a)anthracene	Benzo(b&k)fluoranthene	Benzo(a)pyrene
Benzo(g,h,i)perylene	Bis(2-chloroethyl)ether	Bis(2-chloroethoxy)methane
Bis(2-ethylhexyl)phthalate	Bis(2-chloroisopropyl)ether	4-Bromophenyl phenyl ether
Butyl benzyl phthalate	Carbazole	4-Chloroaniline
1-Chloronaphthalene	2-Chloronaphthalene	4-Chlorophenyl phenyl ether
Chrysene	Dibenzofuran	Dibenzo(a,h)anthracene
Di-n-butylphthalate	1,3-Dichlorobenzene	1,2-Dichlorobenzene
1,4-Dichlorobenzene	3,3'-Dichlorobenzidine	Diethyl phthalate
Dimethyl phthalate	2,4-Dinitrotoluene	2,6-Dinitrotoluene
Di-n-octylphthalate	Fluoranthene	Fluorene
Hexachlorobenzene	Hexachlorobutadiene	Hexachlorocyclopentadiene
Hexachloroethane	Indeno(1,2,3-cd)pyrene	Isophorone
1-Methylnapththalene	2-Methylnaphthalene	Naphthalene
1-Naphthylamine	2-Naphthylamine	2-Nitroaniline
3-Nitroaniline	4-Nitroaniline	Nitrobenzene
N-Nitrosodipheylamine	N-Nitrosodimethylamine	N-Nitrosodi-n-propylamine
N-Nitrosopiperidine	Phenanthrene	Pyrene
Pyridine	1,2,4-Trichlorobenzene	

Acid Extractables:

Benzyl Alcohol	4-Chloro-3-methylphenol	2-Chlorophenol
2,4-Dichlorophenol	2,6-Dichlorophenol	2,4-Dimethylphenol
4,6-Dinitro-2-methylphenol	2,4-Dinitrophenol	2-Methylphenol
3&4-Methylphenol	2-Nitrophenol	4-Nitrophenol
Pentachlorophenol	Phenol	2,4,5-Trichlorophenol
2,4,6-Trichlorophenol		

C. Method 8270 can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate ester, organophosphate ester, nitrosamines, aromatic nitro compounds, and phenols, including nitrophenols.

D. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor. Under the alkaline conditions of the extraction step, alpha-BHC, gamma-BHC, Endosulfan I and II, and Endrin are subject to decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-Nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, 4,6-Dinitro-2-methylphenol, 4-Chloro-3-methylphenol, Benzoic acid, 2-Nitroaniline, 4-Chloroaniline, and Benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

E. The method detection limit (MDL) of Method 8270 ranges from 1.0 to 10ug/L. Practical quantitation limit (PQLs) will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

F. This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

II. Safety:

This method involves the handling of the chemicals outlined below. Each of these chemicals may have specific associated hazards. Analysts must read and become familiar with all elements of each chemical's MSDS. Make special awareness of the symptoms of exposure and potential routes of entry. Analysts must notify the Safety Officer immediately if any symptoms of exposure are suspected.

PPE must be worn at all times in designated work areas. All applicable engineering controls, such as fume hoods or splash shields, must be used when performing analyses. To ensure the safety of all laboratory personnel; work areas must be kept neat and organized, equipment and bench-tops must be cleaned and sterilized, if necessary, at the completion of each task, all containers must be properly labeled as to contents and hazard ratings and spills cleaned immediately and completely.

A. Lab coats, Nitrile Gloves, and Protective Eye Glasses are to be worn at all times during analysis by this method and when handling any laboratory sample.

B. A thorough MSDS review of Methylene Chloride, Sodium Sulfate, Methanol and all laboratory calibration standards should be performed prior to analysis by this method.

C. All standards should be made in fume hood away from heat sources.

D. This method uses reagents that may cause anemia, eye/liver/kidney/central nervous system disorders, reproductive disorders and cancer upon exposure. Stock standards of these materials should be handled in fume hoods. Toxic gas respirators should be worn when handling concentrated standards and samples.

II. Waste Disposal:

A. Methylene Chloride is disposed of in the Methylene Chloride waste satellite carboy and is included with the flammable liquid waste in the Hazardous Waste Room.
B. Dispose of all extracted water waste in the water waste carboy. This is included with the corrosive liquid waste in the hazardous waste room.
C. Dispose of any Solvents in the Misc. Solvent Carboy.
D. Dispose of anything known to contain PCB's to the PCB waste stream in the HazardousWaste Room.
E. Soil samples are disposed of in soil waste drum in Hazardous Waste Room.

III. Sample Collection, Preservation, and Storage:

A. All samples should be refrigerated at 4°C and kept away from light from the time of sampling.
B. pH of water samples are adjusted to between 5 and 9, with 6 N HCl or 6N NaOH.
C. Extraction holding time for water is 7 days and 14 days for soils, from time of sampling, unless the following analytes are to be determined; Carboxin, Diazinon, Disulfoton, Disulfoton sulfoxide, Fenamiphos and Terbufos. These analytes need to be extracted immediately after sampling.
D. Analytical holding time is 30 days after the extraction date.

IV. Equipment:

-Wide mouth Amber Bottles
-1 Liter Amber bottles with Teflon caps
-2L Teflon Seperatory Funnels
-O-Rings and Stands
-Glass Funnels
-pH strips
-500ml Erlenmeyer Flasks
-Shaker Box and Mechanical Shaker
-40ml vial
-Centrifuge
-Zymark Sample Concentrator
-Nitrogen Tank
-Zymark Concentrator Tubes
-2.0 ml Vials
-10ul-1.0 ml glass syringes

-Sample Sonicator
-250 ml Beakers
-Analytical balance, 0.00001 g Accuracy
-Helium Gas
-HP 5890 Series II GC with Restek 30 m RTX-5 Column
-HP 5971 Series Mass Selective Detector
-HP 486 Vectra with monitor and HP G1034C MS ChemStation

V. Reagents:

A. Stock Standard Solutions containing all analytes are purchased from NSI and a duplicate set of analytes is purchased from Accustandard to function as an independent line check and extractable LFB. These standards are replaced on a yearly basis and are stored at -4°C.
B. Internal Standards are purchased from Accustandard (M 8270-IS). The Internal Standards used are 1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12.
C. GC/MS Tuning Standard contains decafluorotriphenylphosphine (DFTPP), 4,4-DDT, Pentachlorophenol and Benzidine purchased from Ultra-Scientific. They are stored at 4°C and replaced annually.
D. Calibration Standards: Standards containing all analytes are made at 11 different concentrations from the Stock standard solutions. The levels are 0.5, 1.0, 2.0, 5.0, 10.0, 20.0, 40.0, 50.0, 60.0, 80.0 and 100.0 ppm. Each standard is then spiked with 40 µg of Internal Standards. These standards are stored at -4°C and replaced yearly or earlier if Independent Line check indicates degradation.
E. Surrogate Standards: Purchased from Accustandard (M-8270-SS) at 4000ppm.
F. Matrix Spike Standards: NSI Custom BNA Spiking Solution (Q-1261-08). Add 400ul of 100ppm Standard.
G. Extraction Solvents
-Methylene Chloride
-Pool Sand--precleaned with Methylene Chloride
-Granular Anhydrous Na_2SO_4
-Methanol - Pesticide Grade
-Florisil – Activated
-Silanated Glass Wool
-DI Water
-10 N NaOH
-H_2SO_4

VI. Interferences:

A. During analysis, major contaminant sources are reagents and phthalates from plastics. Wash all glassware with $MeCl_2$, then MeOH, and then DI Water.
B. Minimize sample contact with plastics. At no time during extraction and analysis are rubber pipette bulbs to be used.

VII. Quality Control: Initial demonstration of Laboratory capability along with an ongoing analysis of Instrument verification and extraction and analysis efficiency samples to evaluate and document data quality. If any data quality issues are not within excepted parameters the procedure is reviewed to determine the source of the problem and corrected. If the problem cannot be corrected the associated analytical data is qualified when reported to the client

A. Initial demonstration of capability (IDC) is performed by analysts training under the supervision of analysts familiar with interpreting GC/MS data and updated annually thereafter. Current IDCs and continuing Demonstration of Capability (DOC) studies are stored in analyst personnel files and are also filed with the Semivolatile QA/QC data. Electronic copies of current DOCs are located on the 8270 computer. (C:/msoffice/excel/qc/semiqc/"year"/idc).
   1. Four replicate mid-calibration reference standards are analyzed by the procedure outlines in section IX
   2. Calculate the average recovery, the standard deviation and the % standard deviation of the recoveries for each analyte of intrest.
   3. Results of IDC are compared to laboratory control charts to see if acceptance criteria are met. In the absence of laboratory acceptance criteria recoveries of 80-120% can be used as guidance in evaluating the results.
   4. If any % standard deviation fails precision limits of 20% or any mean fails accuracy limits (+/-20% or control limit value, whichever is lower), the source of the problem must be found and corrected. Analytes that failed must be reanalyzed and acceptance criteria must be met.

B. Method detection limits (MDL) study is performed annually to determine acceptable detection limits. Current MDL studies are stored with the Semivolatile QA/QC data. Electronic copies of current MDLs are located on the 8270 computer at (C:/msoffice/excel/qc/semiqc/"year"/mdl).
   1. Seven reference standards at 2.5 times the minimum reporting limit are analyzed according to the procedure outlines in section IX
   2. The standard deviation values for each analyte are multiplied by 3.14 to determine the MDL value.
   3. The calculated MDL value must be 2.5 – 5 times less than the target of the reference standards or the MDL is reanalyzed.
   4. The calculated MDL value must be lower than reporting limits outlined in Table 3.

C. Method Reporting Limit determination
   1. Laboratory reporting limit is either the concentration of the lowest analyzed calibration standard or the calculated MDL value for that analyte, whichever value is higher.
   2. Reporting limits can be increased for analytes with poor purge efficiencies, analytes present in the laboratory atmosphere or analytes which tend to carry over from one run to the next as long as the increased reporting limits are lower than minimum requirements for the laboratory regulating bodies (NELAC and State of Vermont).

D. DFTPP analysis performed every 12 hours to confirm that the mass spectrometer is tuned properly for 8270 analysis. The mass intensities are then compared to the tune criteria listed in Table 1.
   1. If all tune criteria is not met, system maintenance and reanalysis of the DFTPP are required until performance is acceptable.
   2. A maximum of 50ng of DFTPP is analyzed at the start of each data set and every 12hours of analysis.
   3. Also included in the mix are Benzidine and Pentachlorophenol to monitor peak shape and total recovery, these compounds are very susceptible to active site degradation and peak tailing.
   4. Also included is 4,4'-DDT which degrades to DDE and DDD, Degradation byproducts must be less than 20% of DDT peak area.

E. Calibration line quality control
   1. The relative standard deviation (RSD) of the initial calibration should be less than 15% for each analyte. Reanalysis of calibration standards and acceptable RSD levels must attained prior to sample analysis.

F. Instrument and software performance
   1. Good column and GC performance should produce symmetrical peaks.
   2. Software should recognize and integrate 99% of compounds analyzed or instrument maintenance is required.
      Entire symmetrical peak for each analyte must elute in the retention time window establish in the initial calibration. Instrument maintenance is require if analytes fall outside retention time windows.

G. Laboratory control standards: Extracted Blanks, Extracted Independent Fortified Blank, and Line Check Standards are performed with each data set.
   1. Extracted Blanks: Analytes from Extracted blanks should be 10 times less than the highest reported value for the data set and below method detection limits to confirm the instrument and laboratory are reasonably free of contamination. System maintenance, removal of laboratory contaminants and additional solvent rinses of glassware on standard re-extraction are required if blank criteria are not met. All data is qualified if blank criteria are not met.

2. Extracted Independent Fortified Standards: Recoveries are tabulated prior to sample analysis. Percent recoveries should fall within acceptable limits tabulated in the method control charts. System maintenance and reanalysis of standards is required prior to sample analysis if data fall out side laboratory control limits. All reported data is qualified if recoveries are outside tabulated acceptance levels.
3. Line Check Standards: Recoveries are tabulated prior to sample analysis. Percent recoveries should fall within acceptable limits tabulated in the method control charts. System maintenance and reanalysis of standards is required prior to sample analysis if data fall out side laboratory control limits. All reported data is qualified if recoveries are outside tabulated acceptance levels.

H. Control charts are tabulated annually from the percent recoveries of the continuous line check and independent fortified standards. Upper and lower warning limits are ± 2 standard deviations of the mean. Upper and lower control limits are ± 3 standard deviations of the mean. Control limits are posted at the 8270 work station and are stored on “8270” at C:msoffice/excel/qc/semiqc/control/”year”.

I. A graphic print out of each analytes recovery is performed annually so recovery trends can be easily viewed.

J. System performance check compounds (SPCC) are performed daily from the line check standard. Minimum acceptable average RFs should be > 0.05 for N-nitroso-di-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 4-Nitrophenol. See calibration and standardization section for RF calculation.

K. Calibration check compounds (CCC) are performed daily from the line check standard. The RF % difference for Acenaphthene, 4-Chloro-3-methylphenol 1,4-Dichlorobenzene, 2,4-Dichlorophenol, Hexachlorobutadiene, 2-Nitrophenol Diphenylamine, Phenol, Di-n-octyl phthalate, Pentachlorophenol, Fluoranthene 2,4,6-Trichlorophenol, and Benzo(a)pyrene should be < 25% of the initial calibration.

$$\% \text{ Difference} = \frac{(\text{RF of Initial Cal} - \text{RF of Current Check std})}{\text{RF of Initial Cal}} \times 100$$

L. Matrix Spikes:Are extracted and analyzed every 20 samples or with each data set if volume or sample mass permits. Percent recoveries are tabulated with the LFB data and are subject to the same acceptance criteria previously stated. If it is determined matrix spike data is outside laboratory control limits due to sample matrix interferences, reported data will be qualified.

M. Duplicate Sample Analysis: Are extracted and analyzed every 20 samples or with each data set if volume or sample mass permits. Analyte percent difference from the MSD must be less than 30% or system maintenance,re-extraction and reanalysis are required. If it is determined duplicate data is outside laboratory control limits due to sample matrix interferences, reported data will be qualified.

N. Evaluation of Retention Times - The relative retention time of each target analyte should agree within 0.06 RRT units

O. Surrogates:
   1. For every sample analyzed the percent recovery of each surrogate is calculated
      a. Once a minimum of thirty samples of the same matrix has been evaluated an Upper and Lower Control Limit for method performance can be generated
   2. If surrogate recovery is not within limits following steps must be taken
      a. Check to be sure there are no errors in calculations
      b. Re-extract and reanalyze the sample or flag the analytes associated with that surrogate as “estimated concentration”
   3. Surrogate recovery data should be updated annually.

P. Peak Integration
   1. Peak integration should be consistent for all analytes, and especially for the integration of standards and samples for the same compound.
      a. Single peaks, the baseline should begin at the start of the upward slope of the peak and end at the bottom of the downward slope.
      b. If there is a raised baseline, the analyst should be careful to not integrate the raised baseline as part of the area of the target peak.
      c. Co-elution of peaks, the baseline should start at the beginning of the upward slope of the peak and remain level until the slope of the valley of the two peaks is at its lowest level. If the target peak is the second of a co-eluting pair, the baseline should be drawn backwards to the same level.

IX. <u>Procedure</u>:

A. Soil Sample Extraction
   1. Place a cleaned glass funnel in a small ring on the ring stand.
   2. Place a wad of glass wool in the bottom of the funnel and fill the funnel with properly conditioned (rinsed) Sodium Sulfate to about 2-3 cm. Be sure to break up any lumps.
   3. Place a precleaned 500 ml-Erlenmyer flask underneath glass funnel.
   4. Rinse the funnel and flask with 20mls of Methylene Chloride and discard the rinse into the Solvent Waste bucket
   5. Repeat Step 4.

6. Weigh 50.0 grams homogeneous sample into a Methylene Chloride rinsed beaker.
7. Label beaker and flask with the Method # and Reference #.
8. Mix in enough Sodium Sulfate with the sample so that the soil is free flowing.
9. Spike the sample with the appropriate compounds.
10. Record the Date, Extractionist's Initials, Reference #, Sample Mass, and Spike Data. (What/How Much/Concentration and Date Opened) in the 8270 log
11. Add 60 ml of solvent to the beaker.
12. Place the sonicator probe in the sample and adjust the sonicator MODE to PULSE, TIME to 180 SEC. and, OUTPUT to about 18%.
13. Sonicate the sample for 180 seconds.
14. Remove the sample from the sonicator and decant the solvent through the sodium sulfate funnel and into the 500ml flask.
15. Repeat steps 11-14 twice.
16. If the Methylene Chloride is still saturated with organic compounds (if the solvent is not clear) repeat steps 11-14 until all of the compounds have been visually extracted and note this change in the 8270 log.
17. After the last of the extract has passed through the sodium sulfate, add 30 mls of solvent to the beaker and then directly decant it down the sides of the Sodium Sulfate funnel.
18. Repeat step 17.
19. Transfer sample into a Methylene Chloride rinsed Zymark Concentrator Flask.
20. Lower flask into Zymark concentrator water bath and concetrated the sample under nitrogen at 45 Degrees Celsius.
21. Continue concentrating the sample until it is about 1-1.5 mls.
22. Transfer the sample from the tube to a 2ml vial with a 2ml Pasteur pipet, and rinse the tube with 3, 300ul aliquots of Methylene Chloride.
23. Determine the volume of the extract with a 0.5ml syringe while transferring it to a new 2.0ml with screw cap. Record the final volume in the 8270 log.
24. Label the vial with Method #, Reference #, Original Volume, Final Volume, Date and Initials. Protect label with Cellophane tape.
25. If the sample is dirty and to be ONLY analyzed for PAHS, follow the clean-up procedure below. Otherwise, continue to Step 26.
26. Store the vials in the Organics Standards fridge in the Hazardous Waste Room until analysis.

B. Full 8270c Water Sample Extraction

1. Place a cleaned glass funnel in a small ring on the ring stand.
2. Place a wad of glass wool in the bottom of the funnel and fill the funnel with properly conditioned (rinsed) Sodium Sulfate to about 2-3 cm. Be sure to break up any lumps.
3. Place a precleaned 500 ml-Erlenmyer flask underneath glass funnel.
4. Rinse the funnel and flask with 20mls of Methylene Chloride and discard the rinse into the Solvent Waste bucket.
5. Repeat Step 4.
6. Label the Erlenmyer flask with Method #, Reference #.
7. Place a 2000ml Separatory Funnel in a large ring above the glass funnel.
8. Rinse the Separatory Funnel with 20mls of Methylene Chloride and discard the rinse into the Solvent Waste bucket.
9. Repeat step 8.
10. If the entire sample is to be used, mark the sample meniscus in the sample bottle and transfer the sample to the Separatory Funnel. If there is soil sediment in the sample, it should not be extracted but it must be noted in the extraction log.
11. Fill the sample bottle with tap water to the meniscus mark and determine the sample volume by transferring the tap water to a 1 liter graduated cylinder.
12. If only a fraction of the sample is to be used, use a clean 100ml graduated cylinder to determine the sample volume.
13. Record the Date, Extractionist's Initials, Reference #, Sample Volume, and general descriptions in the 8270 log.
14. Spike the sample with the appropriate compounds.
15. Use pH paper to check the initial pH and record it in the 8270 log.
16. Raise sample pH > 10 with 10 N NaOH and record it in the log.
17. Add 30 mls of Methylene Chloride to the Separatory Funnel and secure the cap.
18. Shake the sample in the hood for 15 seconds and then hold the Separatory Funnel upside down and open the stopcock to vent the gas.
19. Close the stopcock and put the Funnel in the Shaker box on the Mechanical Shaker and shake the sample for 2 minutes. (4 Funnels must be shaken at the same time.)
20. Place the Funnel back on the stand and allow the emulsion to separate.
21. If the emulsion separates, proceed to step 23, but if it does not separate after 5 minutes, decant it into 40ml vials and then spin the vials in the centrifuge. (DO NOT put more than 30 mls in each 40ml vial)
22. Use a pipette to transfer to Solvent phase to the Sodium Sulfate Funnel and decant the emulsion back into the Separatory Funnel.
23. Drain the lower Methylene Chloride layer from the Separatory Funnel through the Sodium Sulfate funnel into the flasks.
24. Check the pH again and record it in the 8270 log.
25. Repeat steps 17-23 with 60 mls of Methylene Chloride.
26. Repeat steps 17-23 with 30 mls of Methylene Chloride.

27. Lower the sample pH < 2 with Sulfuric Acid and record it in the log book.
28. Repeat steps17-26.
29. After the last of the extract has passed through the sodium sulfate, decant 30 mls of solvent directly decant it down the sides of the Sodium Sulfate funnel.
30. Repeat step 29.
31. Transfer sample into a Methylene Chloride rinsed Zymark Concentrator Flask.
32. Lower flask into Zymark concentrator water bath and concentrated the sample under nitrogen at 45 Degrees Celsius.
33. Continue concentrating the sample until it is about 1-1.5 mls.
34. Transfer the sample from the tube to a 2ml vial with a 2ml Pasteur pipet, and rinse the tube with 3, 300ul aliquots of Methylene Chloride.
35. Determine the volume of the extract with a 0.5ml syringe while transferring it to a new 2.0ml with screw cap. Record the final volume in the 8270 log.
36. Label the vial with Method #, Reference #, Original Volume, Final Volume, Date and Initials. Protect label with Cellophane tape.
37. If the sample is dirty, follow the clean-up procedure below. Otherwise, continue to Step 25.
38. Store the vials in the Organics Standards fridge in the Hazardous Waste Room until analysis.

C. 8270C PAH Water Sample Extraction

1. Place a cleaned glass funnel in a small ring on the ring stand.
2. Place a wad of glass wool in the bottom of the funnel and fill the funnel with properly conditioned (rinsed) Sodium Sulfate to about 2-3 cm. Be sure to break up any lumps.
3. Place a precleaned 500 ml-Erlenmyer flask underneath glass funnel.
4. Rinse the funnel and flask with 20mls of Methylene Chloride and discard the rinse into the Solvent Waste bucket.
5. Repeat Step 4.
6. Label the Erlenmyer flask with Method #, Reference #.
7. Place a 2000ml Separatory Funnel in a large ring above the glass funnel.
8. Rinse the Separatory Funnel with 20mls of Methylene Chloride and discard the rinse into the Solvent Waste bucket.
9. Repeat step 8.
10. If the entire sample is to be used, mark the sample meniscus in the sample bottle and transfer the sample to the Separatory Funnel. If there is soil sediment in the sample, it should not be extracted but it must be noted in the extraction log.
11. Fill the sample bottle with tap water to the meniscus mark and determine the sample volume by transferring the tap water to a 1 liter graduated cylinder.
12. If only a fraction of the sample is to be used, use a clean 100ml graduated cylinder to determine the sample volume.

13. Record the Date, Extractionist's Initials, Reference #, Sample Volume, and general descriptions in the 8270 log.
14. Spike the sample with the appropriate compounds.
15. Use pH paper to check the initial pH and record it in the 8270 log.
16. Add 30 mls of Methylene Chloride to the Separatory Funnel and secure the cap.
17. Shake the sample in the hood for 15 seconds and then hold the Separatory Funnel upside down and open the stopcock to vent the gas.
18. Close the stopcock and put the Funnel in the Shaker box on the Mechanical Shaker and shake the sample for 2 minutes. (4 Funnels must be shaken at the same time.)
19. Place the Funnel back on the stand and allow the emulsion to separate.
20. If the emulsion separates, proceed to step 22, but if it does not separate after 5 minutes, decant it into 40ml vials and then spin the vials in the centrifuge. (DO NOT put more than 30 mls in each 40ml vial)
21. Use a pipette to transfer to Solvent phase to the Sodium Sulfate Funnel and decant the emulsion back into the Separatory Funnel.
22. Drain the lower Methylene Chloride layer from the Separatory Funnel through the Sodium Sulfate funnel into the flasks.
23. Repeat steps 16-22 with 60 mls of Methylene Chloride.
24. Repeat steps 16-22 with 30 mls of Methylene Chloride.
25. After the last of the extract has passed through the sodium sulfate, decant 30 mls of solvent directly decant it down the sides of the Sodium Sulfate funnel.
26. Repeat step 25.
27. Transfer sample into a Methylene Chloride rinsed Zymark Concentrator Flask.
28. Lower flask into Zymark concentrator water bath and concetrated the sample under nitrogen at 45 Degrees Celsius.
29. Continue concentrating the sample until it is about 1-1.5 mls.
30. Transfer the sample from the tube to a 2ml vial with a 2ml Pasteur pipet, and rinse the tube with 3, 300ul aliquots of Methylene Chloride.
31. Determine the volume of the extract with a 0.5ml syringe while transferring it to a new 2.0ml with screw cap. Record the final volume in the 8270 log.
32. Label the vial with Method #, Reference #, Original Volume, Final Volume, Date and Initials. Protect label with Cellophane tape.
33. If the sample is dirty, follow the clean-up procedure below. Otherwise, continue to Step 25.
34. Store the vials in the Organics Standards fridge in the Hazardous Waste Room until analysis.

D. Silica Gel Cleanup for PAH Analysis:
1. Place a large drying column in a clamp on a ring stand.
2. Pack a wad of glass wool down into the tip of a large drying column.
3. Fill the column with 3-4" of silica gel.
4. Rinse the column with 20mls of Methylene Chloride but do not let the column go dry.
5. Once almost all of the Methylene Chloride is through the column place the column in a clean TurboVap Tube and transfer the extract onto the column via pipet.
6. Rinse the old TurboVap tube with 3, 2ml aliquots of Methylene Chloride and transfer these onto the column.
7. Let the solvent flow through the column without applying pressure. This may take awhile.
8. Rinse the column with at least 20mls of Methylene Chloride.
9. Finally, gently blow the last bit of solvent through the column with a large pipet bulb.
10. Repeat the Concentration process and proceed with the rest of the original method.

E. Gas Chromatograph Operating Parameters
1. GC Temperature Zones
   Inj. A: Off
   Inj. B: 280 C
   Det. A: Off
   Det. B: 280 C
2. Oven Parameters
   Oven Equil. Time: 0.40 min
   Oven Max: 315 C
   Oven: On
   Cryo: Off
   Ambient: 25 C
   Cryo Blast: Off
3. Oven Program
   Initial Temp.: 45 C
   Initial Time: 0.70min

Level	Rate (C/min)	Final Temp. C	Final Time (min)
1	4.00	55	0.00
2	8.00	315	6.30
3	0.00		

   Total Run Time: 42.00 min
4. Injector Information
   Injection Source: Auto
   Injection Location: Rear
   Sample Washes: 3
   Sample Pumps: 3

Sample Volume: 2 stops (10 ul Syringe)
Viscosity Delay: 0 sec.
Solvent A Washes: 9
Solvent B. Washes: 9
On Column: No

F. Mass Spectrometer Acquisition Parameters
1. General Information
Inlet: GC
Tune File: DFTPP.U
Acquisition Mode: Scan
2. MS Information
Low Mass: 45
High Mass: 450
Threshold: 120
Sampling #: 2 A/D Samples 4

X. Calibration and Standardization:

A. Generation of 11 Point Line-
Analyze 10 Calibration Standards ranging from 0.5-100 ug; 0.50, 1.0, 2.0, 5.0, 10.0, 20.0, 40.0, 50.0, 60.0, 80.0, 100.

B. Determination of Response Factor:
1. Each analyte or surrogate is associated with its closest Internal Standard.
2. Response factors for each analyte are calculated using the equation:

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

A_x = area of Target Ion of analyte
A_{is} = area of Target Ion of Internal Standard
C_x = Concentration of analyte
C_{is} = Concentration of Internal Standard

C. Refer to Quality Control section for acceptance criteria of Calibration.

XI. Data Interpretation and Data Reduction:

A. All injections made on GC are recorded in “Semi-Volatile GC/MS Log”, including file name, extraction information and volume injected.
B. Qualitative Analysis-Compound identification is determined by satisfying two criteria.
1. Elution of sample compound at the same GC relative retention time as the standard analyte ± 0.06 RRT units.
2. Comparison of sample and standard mass spectrum.

a. All ions greater than 10% of the primary ion in the standard must be present in the sample spectrum.
b. Relative intensities of secondary ions must agree within + or - 20%.
c. Refer to Table 4. For a list of target compounds and their primary & secondary ions.

3. Identification of compounds not in the calibrated standard is determined by comparing to library mass spectra.

C. Quantitation Analysis

1. Quantitation is based on the integrated abundance of the EICP using the Internal Standard Technique.

$$C_x = A_x C_{is} / A_{is} \, RF \, U_o$$

C_x = Concentration of analyte in sample
A_x = Area of EICP of analyte
C_{is} = Amount of ug spiked into sample
A_{is} = Area of EICP of Internal Standard
RF = Average response factor from initial calibration
U_o = Original volume of sample extracted taking into consideration any dilutions

XII. Data Recording:

A. All standards when opened, are recorded in "8270/525 Semi-Volatile Standards Log Book" including date opened, analyst initials, title of standard, manufacturer of standard,lot number and expiration date.

B. All standards prepared in the laboratory should also be recorded in the "8270/525 Semi-Volatile Standards Log Book". Each stock standard used in the making of the standard should be referenced by title and date to when it was opened.

C. A daily run log of all injections made is maintained on a spreadsheet in Excel on the Semi-Volatile computer. The run log should include; date of injection, extraction date,file name, acquisition method, original sample volume, concentration volume, dilution factors, and miscellaneous notes.

D. Sample chromatograms are printed and stored in Reference number sequence with all organic samples. Chromatograms and data for QA/QC are printed and stored in the 8270 QC folder in chronological order.

E. Electronic copies of QC and line checks should be transferred into their corresponding Excel QC spreadsheets.

XIII. Instrument Maintenance/Trouble Shooting:

A. Injection port septa should be changed every 20 injections. Merlin Microseal injection port is good for over 2000 injections. Intermittent integrity checks may be performed by placing flowmeter tube over injection port inlet. If any air flow is measured then the septa is leaking and needs to be chenged.

B. HP service technicians should service instrument, vacuum pump and diffusion pump annually.

C. Injection port should be changed as chromatography deteriorates or as DFTTP ratios worsen. Also removing a 1meter section at the front of the column may improve chromatographic peak shape.

D. Refer to HP 5971 MSD operators manual for source cleaning techniques.

XIV. Method Modifications

XV. Bibliography/Reference

Hewlett Packard Chemstation, Version b.02-02 © Hewlett-Packard 1989-1992

HP 5971A Mass Selective Detector Hardware Manual, edition 1 © Hewlett-Packard Feb. 1990, Publication # 05971-90011

HP 5890A Series II Programmable Cool on Column Inlet, eighth edition © Hewlett-Packard, Sept. 1995 Publication # 05890-90300

HP 5890A Series II Reference Manual, fifth edition © Hewlett-Packard, Sept. 1991Publication # 05890-90270

Method 8000b Determinative Chromatographic Separations, EPA SW-846 NTIS ,CD ROM Version 2, Dec. 1997

Method 8270C Semivolatile Organic Compounds by GC/MS. Revision 3. US EPA. December 1996,

XVI. REVISION HISTORY

Revision #7. April 6, 2005

-Revised safety section
-Updated QC section
-Added Table #3 – Detection Limits
-Added Bibliography
-Added Revision History
-Added sign off sheet

TABLE 1. DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

[a]J.W. Eichelberger, L.E. Harris, and W.L. Budde. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry", Analytical Chemistry, 47, 995 (1975).

TABLE 2. CALIBRATION CHECK COMPOUNDS (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 3.

Compound	Reporting Limit Waterug/L	Reporting Limit Soilug/Kg	Compound	Reporting Limit Waterug/L	Reporting Limit Soilug/Kg
Acenaphthene-d (IS)	NA	NA	2,4-Dinitrophenol	20.0	400.
Acenaphthene	2.0	40.0	2,6-Dinitrophenol	20.0	400.
Acenaphthylene	2.0	40.0	2,4-Dinitrotoluene	5.0	100.
Aniline	5.0	100.	2,6-Dinitrotoluene	5.0	100.
Anthracene	2.0	40.0	Fluoranthene	2.0	40.0
Benz(a)anthracene	2.0	40.0	Fluorene	2.0	40.0
Benzidine	15.0	300.	Hexachlorobenzene	5.0	100.
Benzo(a)pyrene	2.0	40.0	Hexachlorobutadiene	5.0	100.
Benzo(b)fluoranthene	2.0	40.0	Hexachlorocyclopentadiene	20.0	400.
Benzo(k)fluoranthene	2.0	40.0	Hexachloroethane	5.0	100.
Benzo(g,h,i)perylene	2.0	40.0	Indeno(1,2,3-cd)pyrene	2.0	40.0
Benzyl alcohol	10.0	200.	Isophorone	2.0	40.0
Bis(2-chloroethoxy)methane	5.0	100.	1-Methylnaphthalene	2.0	100.
Bis(2-chloroethyl) ether	5.0	100.	2-Methylnaphthalene	2.0	100.
Bis(2-chloroisopropyl) ether	10.0	200.	2-Methylphenol	5.0	100.
Bis(2-ethylhexyl) phthalate	5.0	100.	3-Methylphenol	5.0	100.
4-Bromophenyl phenyl ether	2.0	40.0	4-Methylphenol	5.0	100.
Butyl benzyl phthalate	5.0	100.	Naphthalene-d (IS)	NA	NA
Carbazole	10.0	200.	Naphthalene	2.0	40.0
4-Chloroaniline	5.0	100.	1-Naphthylamine	10.0	200.
4-Chloro-3-methylphenol	10.0	200.	2-Naphthylamine	10.0	200.
1-Chloronaphthalene	2.0	40.0	2-Nitroaniline	20.0	400.
2-Chloronaphthalene	2.0	40.0	3-Nitroaniline	10.0	200.
2-Chlorophenol	5.0	100.	4-Nitroaniline	20.0	400.
4-Chlorophenyl phenyl ether	2.0	40.0	Nitrobenzene	2.0	40.0
Chrysene	2.0	40.0	2-Nitrophenol	10.0	200.
Chrysene-d (IS)	NA	NA	4-Nitrophenol	10.0	200.
Dibenz(a,h)anthracene	2.0	40.0	N-Nitrosodi-n-butylamine	5.0	100.
Dibenzofuran	2.0	40.0	N-Nitrosodi-n-propylamine	10.0	200.
1,2-Dichlorobenzene	2.0	40.0	N-Nitrosodiphenylamine	5.0	100.
1,3-Dichlorobenzene	2.0	40.0	N-Nitrosopiperidine	10.0	200.
1,4-Dichlorobenzene-d (IS)	NA	NA	N-Nitrosodimethylamine	5.0	100.
1,4-Dichlorobenzene	2.0	40.0	Pentachlorophenol	15.0	300.
3,3'-Dichlorobenzidine	5.0	100.	Perylene-d (IS)	NA	NA
2,4-Dichlorophenol	5.0	100.	Phenanthrene	2.0	40.0
Di-n-butyl phthalate	5.0	100.	Phenanthrene-d (IS)	NA	NA
Di-n-octyl phthalate	5.0	100.	Phenol	5.0	100.
Diethyl phthalate	5.0	100.	Pyrene	2.0	40.0
2,4-Dimethylphenol	5.0	100.	1,2,4-Trichlorobenzene	2.0	40.0
Dimethyl phthalate	5.0	100.	2,4,5-Trichlorophenol	10.0	200.
4,6-Dinitro-2-methylphenol	50.0	2,000.	2,4,6-Trichlorophenol	10.0	200.

TABLE 4.

Compound	Primary Ion	Confirmation Ion(s)
Acenaphthene-d (IS)	164	162,160
Acenaphthene	154	153,152
Acenaphthylene	152	151,153
4-Aminobiphenyl	169	168,170,115
Aniline	93	66,65
Anthracene	178	176,179
Benz(a)anthracene	228	229,226
Benzidine	184	92,185
Benzoic acid	122	105,77
Benzo(a)pyrene	252	253,125
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(g,h,i)perylene	276	138,277
Benzyl alcohol	108	79,77
Bis(2-chloroethoxy)methane	93	95,123
Bis(2-chloroethyl) ether	93	63,95
Bis(2-chloroisopropyl) ether	45	77,121
Bis(2-ethylhexyl) phthalate	149	167,279
4-Bromophenyl phenyl ether	248	250,141
Butyl benzyl phthalate	149	91,206
Carbofuran	164	149,131,122
4-Chloroaniline	127	129,65,92
4-Chloro-3-methylphenol	107	144,142
1-Chloronaphthalene	162	127,164
2-Chloronaphthalene	162	127,164
2-Chlorophenol	128	64,130
4-Chlorophenyl phenyl ether	204	206,141
Chrysene	228	226,229
Chrysene-d (IS)	240	120,236 12
Dibenz(a,h)anthracene	278	139,279
Dibenzofuran	168	139
1,2-Dichlorobenzene	146	148,111
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene-d (IS)	152	150,115
1,4-Dichlorobenzene	146	148,111
3,3'-Dichlorobenzidine	252	254,126
2,4-Dichlorophenol	162	164,98
Di-n-butyl phthalate	149	150,104
Di-n-octyl phthalate	149	167,43
Diethyl phthalate	149	177,150
2,4-Dimethylphenol	122	107,121
Dimethyl phthalate	163	194,164
1,4-Dinitrobenzene	168	75,50,76,92,122
1,3-Dinitrobenzene	168	76,50,75,92,122
1,2-Dinitrobenzene	168	50,63,74
4,6-Dinitro-2-methylphenol	198	51,105
2,4-Dinitrophenol	184	63,154

TABLE 4. cont.

Compound	Primary Ion	Confirmation Ion(s)
2,6-Dinitrophenol	162	164,126,98,63
2,4-Dinitrotoluene	165	63,89
2,6-Dinitrotoluene	165	63,89
Diphenylamine	169	168,167
Fluoranthene	202	101,203
Fluorene	166	165,167
Hexachlorobenzene	284	142,249
Hexachlorobutadiene	225	223,227
Hexachlorocyclopentadiene	237	235,272
Hexachloroethane	117	201,199
Indeno(1,2,3-cd)pyrene	276	138,227
Isophorone	82	95,138
1-Methylnaphthalene	142	141
2-Methylnaphthalene	142	141
2-Methylphenol	107	108,77,79,90
3-Methylphenol	107	108,77,79,90
4-Methylphenol	107	108,77,79,90
Naphthalene-d (IS)	136	68
Naphthalene	128	129,127
1-Naphthylamine	143	115,89,63
2-Naphthylamine	143	115,116
2-Nitroaniline	65	92,138
3-Nitroaniline	138	108,92
4-Nitroaniline	138	65,108,92,80,39
Nitrobenzene	77	123,65
2-Nitrophenol	139	109,65
4-Nitrophenol	139	109,65
N-Nitrosodiethylamine	102	42,57,44,56
N-Nitrosodi-n-butylamine	84	57,41,116,158
N-Nitrosodi-n-propylamine	70	42,101,130
N-Nitrosodiphenylamine	169	168,167
N-Nitrosopiperidine	114	42,55,56,41
N-Nitrosopyrrolidine	100	41,42,68,69
Pentachlorophenol	266	264,268
Perylene-d (IS)	264	260,265 12
Phenanthrene	178	179,176
Phenanthrene-d (IS)	188	94,80 10
Phenol	94	65,66
Pyrene	202	200,203
1,2,4-Trichlorobenzene	180	182,145
2,4,5-Trichlorophenol	196	198,97,132,99
2,4,6-Trichlorophenol	196	198,200

I acknowledge that I have read this Standard Operating Procedure and agree to adhere to the Safety and Quality Control policies outlined within.

Name	Date

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ORGANOCHLORINE PESTICIDES
Method EPA 8081A

Standard Operating Procedure

REVISED: November 6, 2006

Revision #: 9

Technical Director: _____Date :_____
Harry B. Locker Ph.D.

QA Officer: _____Date :_____
Mark L. Fausel

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I. Scope and Application

A. Overview

EPA Method 8081A is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices. Samples are extracted by appropriate EPA 3500 series methods, concentrated to 4.0-mL using a Zymark TurboVap II workstation and cleaned using EPA 3600series methods. The extract is analyzed by GC/ECD using Restek Rtx-CLPesticides (or equivalent) capillary column. The compounds listed below are determined by a single column analysis system and confirmed on a Restek Rtx-CLPesticides II (or equivalent) capillary column.

1. Compounds and Reporting Limits

- Additional compounds not listed below may also be determined using this method.

Compound	Water	Soil	TCLP
alpha-BHC	< 0.1 µg/L	< 5.0µg/kg	---
beta-BHC	< 0.1 µg/L	< 5.0µg/kg	---
gamma-BHC (Lindane)	< 0.1 µg/L	< 5.0µg/kg	< 2.0 µg/L
delta-BHC	< 0.1 µg/L	< 5.0µg/kg	---
Heptaclor	< 0.1 µg/L	< 5.0µg/kg	< 2.0 µg/L
Aldrin	< 0.1 µg/L	< 5.0µg/kg	---
Hetachlor Epoxide	< 0.1 µg/L	< 5.0µg/kg	< 2.0 µg/L
Endosulfan I	< 0.1 µg/L	< 5.0µg/kg	---
4,4’-DDE	< 0.1 µg/L	< 5.0µg/kg	---
Dieldrin	< 0.1 µg/L	< 5.0µg/kg	---
Endrin	< 0.1 µg/L	< 5.0µg/kg	< 2.0 µg/L
Endosulfan II	< 0.1 µg/L	< 5.0µg/kg	---
4,4’-DDD	< 0.1 µg/L	< 5.0µg/kg	---
Endrin Aldehyde	< 0.1 µg/L	< 5.0µg/kg	---
Endosulfan Sulfate	< 0.1 µg/L	< 5.0µg/kg	---
4,4’-DDT	< 0.1 µg/L	< 5.0µg/kg	---
Methoxychlor	< 0.1 µg/L	< 5.0µg/kg	< 20.0 µg/L
Chlordane	< 0.5 µg/L	< 20.0µg/kg	< 10.0 µg/L
Toxaphene	< 0.5 µg/L	< 20.0µg/kg	< 40.0 µg/L

2. The analysis of PCBs should be undertaken using method 8082, which includes specific cleanup and quantitation procedures designed for PCB analysis. If both the analysis of organochlorine pesticides and PCBs are requested for a particular sample, split the extract prior to any cleanup steps and process part of the extract for organochlorine pesticide analysis and the other portion for PCB analysis using Method 8082.

3. Matrix specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.).

4. When samples contain more than one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation.

5. Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique.

6. This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

B. Summary of Method

1. A measured volume or weight of sample (approximately 1-L for liquids, 2g – 30g for solids and 50mL for TCLP) is extracted using the appropriate matrix-specific sample extraction technique.

a) Liquid samples are extracted at a neutral pH (pH 5-9) with methylene chloride using Method 3510 (separatory funnel) or Method 3520 (continuous liquid-liquid extractor).

b) Solid samples are extracted with 1:1 acetone/methylene chloride using Method 3550 (ultrasonic extraction) or with 1:1 acetone/hexane using Method 3540 (soxhlet extraction).

c) A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interference and the target analytes. Sulfur cleanup with copper powder (Method 3660) is currently the only cleanup technique in use, but alumina (Method 3610), Florisil (Method 3620), silica gel (Method 3630) and gel permeation chromatography (Method 3640) are other cleanups specified by this method.

2. After cleanup, the extract is analyzed by injecting a 2-uL sample into a gas chromatograph with a fused silica capillary column and electron capture detector (GC/ECD).

II. Safety

1. This method involves the handling of the chemicals outlined below. Each of these chemicals may have specific associated hazards. Analysts must read and become familiar with all elements of each chemical's MSDS. Make special awareness of the symptoms of exposure and potential routes of entry. Analysts must notify the Safety Officer immediately if any symptoms of exposure are

suspected.

2. PPE must be worn at all times in designated work areas. All applicable engineering controls, such as fume hoods or splash shields, must be used when performing analyses. To ensure the safety of all laboratory personnel; work areas must be kept neat and organized, equipment and bench-tops must be cleaned and sterilized, if necessary, at the completion of each task, all containers must be properly labeled as to contents and hazard ratings and spills cleaned immediately and completely.

3. Lab coats, Nitrile Gloves, and Protective Eyeglasses are to be worn at all times during analysis by this method and when handling any laboratory sample.

4. A thorough MSDS review of Methylene Chloride, Sodium Sulfate, Methanol, Hexane and all laboratory calibration standards should be performed prior to analysis by this method.

5. All standards should be made in fume hood away from heat sources.

6. This method uses reagents that may cause anemia, eye/liver/kidney/central nervous system disorders, reproductive disorders and cancer upon exposure. Stock standards of these materials should be handled in fume hoods. Toxic gas respirators should be worn when handling concentrated standards and samples.

III. Waste Management:

A. Solvent Waste:

All solvent waste must be disposed of in the solvent waste satellite container, then placed in the larger container located in the hazardous waste room. Note: Dumping of solvent waste in the Hazardous waste room can ONLY be done by a Technician certified to do so.

B. Water/Soil Waste:

Solid and aqueous material must sit in a vented area until all the solvent has been evaporated. Alternatively, it may be collected in a carboy and bubbled with the exhaust of the pump in the extraction lab hood. The water can be flushed down the drain with copious amount of tap water. Soil waste is stored in a satellite container and disposed of in the solid waste container in the hazardous waste room.

C. Acid Waste:

Acid waste should be contained separate from all other waste streams and disposed of in the proper waste container in the hazardous waste room by a technician who is trained to do so.

IV. Sample Collection, Preservation, and Storage:

A. Sample Collection and Preservation

1. Collect water samples in 1L amber glass and preserve with Sodium Thiosulfate. (Sodium Thiosulfate is used when Aldrin is to be determined and when residual chlorine is present)
2. Adjust the pH to 5.0 - 9.0, using 1:1 H_2SO_4 or 10N NaOH as needed, if the sample is not going to be extracted within 72 hours.
    a) Record the volumes added on the Chain-of-Custody and store the preserved sample in a 4^oC refrigerator.
3. Collect solids in 250mL amber glass, and refrigerate at 4^oC.

B. Storage

1. All samples are stored in the Extraction Lab refrigerator at 4^oC.
2. All extracts are stored in the Sample Extract refrigerator in the extraction lab at 4^oC.
3. Standards are stored in the Extraction Lab and Organics Lab Standard refrigerators at 4^oC.

C. Holding Times

1. Extraction
    a) Waters: 7 days from sampling
    b) Solids: 14 days from sampling
2. Analysis
    a) 40 days from extraction

V. Equipment and Supplies

A. Analysis

1. Desktop computer running HP Chemstation 1701 BA
2. Hewlett-Packard 5890 GC
3. Hewlett-Packard 19233 Electron Capture Detector
4. Hewlett-Packard 7673 Autosampler
5. Rtx-CLPesticides (or equivalent) column: 30m, 0.32mm Id, 0.5um film – primary analysis.
6. Rtx-CLPesticides II (or equivalent) column: 30m, 0.32mm Id, 0.25um film – confirmation analysis.
7. Merlin microseal – septa
8. Cyclosplitter injection port liner (or equivalent)
9. J&W Electronic Flow Meter
10. Vent Carbon Trap

11. Ultra-High Purity N_2 gas supply

B. Extraction

1. 375 watt Ultrasonic Disrupter (*Tekmar*)
   a) ¾" horn (low concentration)
   b) 1/8" tapered microtip attached to a ½" horn (high concentration)
2. 250mL Beakers
3. 500mL Erlenmeyer Flasks
4. Glass funnels
5. Various sized syringes
6. 2 Liter *Teflon* separatory funnels
7. 1 Liter graduated cylinder
8. 9" Glass Pasteur pipettes
9. 2mL and 4mL vials, *Teflon* septa caps, and micro inserts
10. Metal spatulas
11. *Zymark* TurboVap II concentrator
12. *Zymark* TurboVap II concentration vials
13. Soxhlet extractors with 500mL round bottom flasks
14. Continuous liquid-liquid extractor with 500mL round bottom flask
15. Heating mantle – Rheostat controlled
16. Boiling chips – solvent rinsed

VI. Reagents and Standards:

A. Reagents Log

1. All reagents are recorded in the Reagents Log when opened. The date reagents are entered into the Reagents Log is then used to trace reagents on the Extraction Log.

B. Reagents

1. Reagent grade chemicals that conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.
2. Hexane – Fisher Optima
3. Acetone – Fisher Optima
4. Methylene chloride – GC Resolv
5. Methanol
6. Anhydrous granular sodium sulfate – Certified ACS
   a) Purify by heating to 400 ^{o}C for 4 hours in a shallow tray.
   b) The sodium sulfate log is kept by the analyst who is responsible for baking the sodium sulfate in the muffle furnace. Reagent information as

well as baking information (time, temperature…) is recorded here.

7. De-ionized water – produced by Endyne, Inc
8. Pool sand – Methylene chloride rinsed
9. Glass wool – Silanated
10. 1:1 Sulfuric acid
11. 10 N NaOH
12. Nitric acid – dilute
13. Copper powder – remove oxides by treating with dilute nitric acid, rinse with DI to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen.

C. Standards

1. Store standards at 4 ^{o}C in PTFE-sealed containers in the standards refrigerator in the extraction lab.
2. Ensure that all purchased standards are in hexane, acetone or solvents compatible with hexane.
3. Standards purchased for QCS samples should come from a separate supplier than calibration standards – when possible.
4. AccuStandard M-8080 (2000ppm Pesticides Mix) or equivalent – 2 lots, one for calibration and one for QC.
5. AccuStandard CLP-032-R (200ppm decachlorobiphenyl and tetrachloro-m-xylene) or equivalent
6. AccuStandard P-093S-1 (200ppm Toxaphene) or equivalent – 2 lots
7. AccuStandard P-017S-1 (200ppm Chlordane) or equivalent – 2 lots

VII. Interferences:

A. Sources

1. Contaminated solvents, reagents, or sample processing hardware.
2. Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.
3. Compounds extracted from the sample matrix to which the detector will respond.

B. Prevention and Solutions

1. Phthalate esters from flexible plastics are introduced during sample preparation and can pose a major problem in pesticide determinations. Method 3640 or Method 3630 may be employed to remove these compounds, but precautions should be taken not to contaminate glassware, solvents or reagents used in this extraction.
2. Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and DI water. Drain the glassware and dry it in an oven at 130 ^{o}C for several hours or rinse with MeOH and drain. Volumetric glassware should not be subjected to heating.

Store dry glassware in a clean environment.

3. Elemental sulfur should be cleaned up using Method 3660. Since Method 3660 reduces the recovery of endrin aldehyde, this compound must be determined prior to sulfur cleanup.

4. Waxes, lipids, and other high molecular weight materials can be removed by Method 3640.

5. PCBs, other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Cleanup methods 3630, 3620 and 3610 may be employed to deal with these co-elution problems. Original methods should be consulted for more detail.

VIII. Quality Control:

A. Extraction

1. The following should be extracted at least 1 per set or 1 per 20 samples, whichever is more frequent (for TCLP samples, LRB and LFB samples will use 50mL TCLP extraction fluid #2 and 950mL of DI):
2. Laboratory Reagent Blank (LRB)
   a) 1.0L DI H_2O spiked with 20uL of 25ppm TCMX &DCB
3. Laboratory Fortified Blank (LFB)/Quality Control Sample (QCS)
   a) 1.0L DI H_2O spiked with 20uL of 25ppm TCMX & DCB, and 10uL of 50ppm Pesticide QC mix.
   b) The stock standard pesticide mix should come from a source independent from the calibration stock standard.
4. Laboratory Duplicate (LD)
   a) A sample aliquot taken in addition to the first, spiked with 20uL of 25ppm TCMX & DCB, extracted, and analyzed with an identical procedure to the first in order to measure the precision associated with this laboratory procedure.
5. Laboratory Fortified Sample Matrix (LFM)
   a) A sample aliquot taken in addition to the first, and spiked the same as an LFB to determine whether the sample matrix contributes bias to the analytical results.

B. Method Blanks/LRB

1. When samples that are extracted together are analyzed on separate instruments or on separate analytical shifts, the method blank must be analyzed on at least one of those instruments.
2. The method blank may be analyzed immediately after the calibration verification standard, to ensure that there is no carryover from the standard, or at another point in the analytical shift.
3. When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.
4. LRBs should have target compounds:
   a) Less than the MDL for the analyte;

b) Less than 5% of the regulatory limit associated with an analyte;
c) Less than 5% of the sample result for the same analyte, whichever is greater.

5. If the method blank results do not meet the acceptance criteria above, then the laboratory should take corrective action to locate and reduce the source of the contamination and to re-extract and reanalyze any samples associated with the contaminated method blank.
6. If method blank does not meet acceptance criteria and samples are past holding times, reported data will be qualified "B" indicating that blank contamination was observed at levels affecting analytical results.
7. Blank subtraction is not permitted.
8. Method blanks and/or solvent blanks may be used to check for contamination by carryover from a high-concentration sample into subsequent samples.
9. When analysis of such blanks is not possible the analyst should review the result for at least the next two samples after the high-concentration sample.
   a) If the analytes in the high-concentration sample are not present in subsequent samples, the lack of carryover has been demonstrated.
   b) If there is evidence that carryover may have occured, then the affected samples should be reanalyzed.

C. LFB/QCS sample

1. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. It is fortified at the same concentration as the matrix spike.
2. Recovery calculations are as follows: %Recovery = ((Cs – Cu)/ Cn)*100 where:
   a) Cs = Measured concentration of the spiked sample aliquot
   b) Cu = Measured concentration of the unspiked sample aliquot (use 0 for the LCS)
   c) Cn = Nominal (theoretical) concentration increase that results from spiking the sample or the nominal concentration of the spiked aliquot (for the LCS)
3. In the absence of control limits, 70-130% may be used.
4. If the LCS falls outside of acceptance limits, the laboratory should take the following corrective action:
   a) Check to be sure that there are no errors in the calculations or standard solutions. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.
   b) Check instrument performance. If an instrument performance problem is found, correct the problem and re-analyze the extracts as well as all associated sample extracts.
   c) If no instrument problem is found, the analytical batch should be re-extracted and re-analyzed if still within holding time.
   d) If upon re-analysis (of initial extract or re-extracted extract), the recovery is again not within limits, affected analytes should be qualified

"QA" indicating that associated QA/QC did not meet laboratory guidelines.

D. Matrix Spike (MS)/LFM sample

1. A duplicate field sample is fortified with a known amount of analyte in order to document the effect of the matrix on method performance.
2. Matrix spikes are fortified with the same amount of analyte as the LFB/LCS.
3. Matrix Spike recoveries are not calculated if the fortified concentration is <20% of the background concentration of the sample.
4. Recoveries for Matrix Spike samples are calculated in the same manner as the LFB/LCS and must meet established control limits.
5. In the absence of control limits, 70-130% may be used.
6. When the results of a matrix spike analysis indicates a potential problem due to the sample matrix itself (a positive of negative bias) the LFB/LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
7. If it is determined that the matrix is biasing quantitation, affected analytes are to be qualified "M+" or "M-" indicating a positive or negative bias due to the sample matrix.
8. If both the LCS and MS fall outside of acceptance limits, follow the corrective action outlined for the LFB/QCS (sec. VII.C.4).

E. Duplicate/LD

1. A duplicate sample is analyzed along side the sample in order to measure the precision associated with this method.
2. Precision is an estimate of the relative percent difference (RPD) of the concentrations for the analysis of the unspiked samples.
3. RPD = ((C1-C2)/((C1+C2)/2))*100 where:
   a) C1 = measured concentration of the first sample aliquot
   b) C2 = measured concentration of the second sample aliquot
4. Acceptance limits for precision are 70-130% RPD
5. Duplicates that fall outside acceptance criteria should be qualified as "DS," indicating that there were disparate results between laboratory duplicate analyses.

F. Surrogates

1. In addition to the fortification of all QA/QC samples with 20uL of 25ppm TCMX &DCB surrogate mixture, all field samples must also be fortified and evaluated versus in-house acceptance criteria.
2. Surrogate recoveries is calculated as: (Recovered Surrogate Concentration / Surrogate Target Concentration) * 100
3. Surrogate recoveries must fall within established acceptance limits. If surrogate recoveries are outside of acceptance limits the following steps should be taken.
   a) Check to be sure that there are no errors in the calculations or surrogate standard solutions. If errors are found, recalculate the data

accordingly. Examine chromatograms for interfering peaks and integrated peak areas.

b) Check instrument performance. If an instrument performance problem is found, correct the problem and re-analyze the extract.

c) Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analysis should be provided to the data user.

d) If no instrument problem is found, the sample should be re-extracted and re-analyzed if still within holding time.

e) If upon re-analysis (of initial extract or re-extracted extract), the recovery is again not within limits, Surrogate value reported to the client must be qualified as "NR" indicating that the surrogate falls outside laboratory QA/QC parameters.

G. Demonstration of Capability (DOC)

1. Prior to extracting samples for clientele, a new extractionist must extract 4 LFB samples at the standard QC concentration. These LFB must be analyzed as other laboratory samples and then processed according to the following acceptance criteria:

a) Calculate the average in ug/L and the standard deviation for each parameter using the four results

b) The average % Recovery must fall within established control limits for LFBs, and the percent standard deviation must be less than 20%.

H. Method Detection Limits (MDL)/Reporting Limits

1. An MDL study should be performed once a year.

a) Seven low-level QCs are extracted. To determine the MDL for each analyte and surrogate, the standard deviation of the seven values is multiplied by 3.14.

b) The target concentration of the low-level QCs should be 2.5-5 times the calculated MDL value.

c) Method Detection Limit should be no greater than one-fifth the Reporting Limit.

2. The reporting limit is based on achieving the requested detection limits of our clients as well as being below known environmental action limits. The laboratory takes into account known interferences, such as contamination peaks. It should be no less than 5 times the MDL.

3. For established reporting limits refer to Section I.

I. Limits, Tables, and Charts

1. Tables of surrogate and analyte recoveries from LFBs and LFMs are maintained, and updated annually. Surrogate data is established from field samples and LFB and LFM data come from LFBs and Matrix Spike samples.
2. Limits are based on the previous chronological year's data, unless significant modification has been made to the methodology in which case limits are established after the first twenty samples have been analyzed.
   a) Control Limits: parameter average +/- 3(Standard Deviation)
   b) Warning Limits: parameter average +/- 2(Standard Deviation)
   c) Control Charts must be separated by matrix and extraction procedure (Ex: Soil by Sonication, Soil by Soxhlet, Soil by Continuous)
   d) Up to date Control Charts are kept in the 608 analyst's workspace.

J. Retention Time Window

1. Retention Time Windows (RTW) are used to determine whether or not a target analyte is present in a sample.
   a) Establish a RTW by first making three (3) injections of calibration standard over a 72-hour period.
   b) Record the RT's for each component to the third decimal place.
   c) Calculate the mean and standard deviation for each component.
   d) For multi-peaked compounds, choose 4-6 of the biggest peaks
   e) RTW Width: +/- 3(STDEV) of the absolute RT average OR 0.03, whichever is greater.
   f) The experience of the analyst should weigh heavily in the interpretation of chromatograms.
2. On subsequent analyses of samples, establish RT's from the first calibration line check.
3. For samples run with the initial calibration, the middle line point is used as the RT reference.
4. RTW must be established for each column used to analyzed samples.

K. Qualifying Data

1. Any deviations from the above outlined quality control as well as any sample container, preservation or holding time discrepancies, outlined in Section IV, are noted on the final report.

IX. Procedures

A. Extraction Log

1. An 8080/608 Extraction Log is to be filled out for every extraction batch and kept in the extraction log binder in the extraction lab. Copies of the extraction log are to be stapled to the Login Reports and given to the analyst when extractions have been completed.

B. Separatory Funnel Liquid-Liquid Extraction (SW-846 Method 3510C)

1. *Pre-rinse all extraction apparatus with $MeCl_2$ before use.
2. Using a 1-L graduated cylinder, measure 1-L of sample. Alternatively, if the entire contents of the sample bottle are to be extracted, mark the level of the sample on the outside of the bottle. If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1-L with DI. TCLP samples require that 50mL of sample be diluted to 1.0L with DI.
    a) 1-L of DI H_2O is to be used for the blank and QC sample.
    b) When TCLP samples are being extracted, both the blank and QC should use 50mL of TCLP extraction fluid #2 (the same batch used to extract the samples) and 950mL of DI.
3. Spike 20ul of the 25ppm surrogate spiking solution into each sample (including blanks, QC samples and matrix spike samples) in the graduated cylinder (or sample bottle) and mix well.
    a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 10ul of the 50ppm 8081A Pesticides QC standard.
4. Check the pH of the sample with wide-range pH paper to ensure a neutral pH between 5 and 9.
5. Quantitatively transfer the sample from the graduated cylinder (or sample bottle) to the 2-L separatory funnel. Use 60mL of methylene chloride to rinse the cylinder (or bottle) and transfer this rinse solvent to the separatory funnel.
    a) If the sample was transferred directly from the sample bottle, refill to the mark made in Sec. X.B.2 with water and then measure the volume of sample that was in the bottle.
6. Seal and shake the separatory funnel vigorously for 1- 2 minutes with periodic venting to release excess pressure. The automatic shaker may be employed on the high setting for 2 minutes after the separatory funnel has been shaken by hand and vented periodically for 30 seconds.
    a) Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. The separatory funnel should be vented into a hood to avoid exposure of the analyst to solvent vapors.
7. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques.
    a) The optimum technique depends on the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.
8. Decant the solvent extract through a glass funnel containing sodium sulfate and into a 500mL Erlenmeyer flask.
    a) Plug a glass funnel with glass wool and add sodium sulfate until it is 2-3cm above the glass wool plug.
9. Repeat the extraction (steps 5-8) two more times adding fresh portions of

methylene chloride directly to the separatory funnel. On the final extraction, rinse the sodium sulfate with two 30ml portions of methylene chloride.
10. Perform the concentration using the TurboVap Concentration Method (Sec. IX.G)

C. Ultrasonic Extraction (SW-846 Method 3550B)-Low Concentration, <20mg/kg

1. The following steps should be performed rapidly to avoid the loss of the more volatile extractable compounds.
2. *Pre-rinse all extraction apparatus with methylene chloride before use.
3. Weigh approximately 30g of sample into a 500ml beaker. Record the weight to the nearest 0.01g. 30g of methylene chloride rinsed pool sand are to be used for the blank and QC sample.
    a) Samples must be well mixed prior to weighing. Dump entire contents of sample into a tared beaker, remove any foreign objects (sticks, leaves and rocks) and thoroughly mix the sample with a spatula. Replace sample into original sample container leaving 30g in the beaker to be extracted.
4. Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture must be mixed with 60g of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate may be added.
    a) After addition of sodium sulfate, the sample should be free flowing.
5. Spike 20ul of the 25ppm surrogate standard solution onto all samples, matrix spike samples QC samples and blanks.
    a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 10ul of the 50ppm 8081A Pesticides QC standard.
6. Immediately add 100mL of 1:1 acetone/methylene chloride.
7. Tune sonicator daily or after changing the horn (use ¾ inch horn).
    a) Ensure that the probe is not immersed in liquid and that it does not come in contact with anything.
    b) Set PULSER to OFF.
    c) Set OUTPUT CONTROL to “10”
    d) Set POWER SWITCH to ON. The switch will illuminate.
    e) Momentarily depress TUNE SWITCH and rotate the tuner clockwise or counterclockwise until a minimum (not maximum) reading (usually <20) is obtained on power monitor.
    f) Set OUTPUT CONTROL to “10”
    g) Set % DUTY CYCLE to “50”
    h) Set TIMER to “180S”
    i) Set PULSER to ON position
8. Before each sample, place the probe tip in fresh 1:1 acetone/methylene chloride and energize the power supply for a few seconds to remove any residue.
9. Place the bottom surface of the tip of the ¾ inch disrupter horn about ½ inch below the surface of the solvent, but above the sediment layer. If the horn comes in contact with the sediment output is reduced.
10. Press START on sonicator and extract ultrasonically for 3 minutes, with

output control knob set to 10 (full power) at 50% duty cycle. Do not use microtip probe.

a) *It is highly advisable to wear earplugs during the sonication process to prevent possible damage to one's hearing.

11. Decant the solvent extract through a glass funnel containing sodium sulfate and into a 500mL Erlenmeyer flask.

a) Plug a glass funnel with glass wool and add sodium sulfate until it is 2-3cm above the glass wool plug.

12. Repeat the extraction two or more times with two additional 100mL portions of 1:1 acetone/methylene chloride. Decant off the solvent after each ultrasonic extraction. On the final ultrasonic extraction, pour the entire sample onto the sodium sulfate and rinse with two 50ml portions of 1:1 acetone/methylene chloride.

13. Perform the concentration using the TurboVap Concentration Method (Sec. IX.G)

D. Ultrasonic Extraction (SW-846 Method 3550B)-High Concentration, >20mg/kg

1. The following steps should be performed rapidly to avoid the loss of the more volatile extractable compounds.
2. Transfer approximately 2g (record weight to the nearest 0.01g) of well-mixed sample to a 40mL VOA vial. Wipe the mouth of the vial with a Kimwipe to remove any sample material. Record the exact weight of the sample taken.
3. Add 2g anhydrous sodium sulfate to the sample in the 40mL vial and mix well.
4. Spike 50ul of the 25ppm surrogate standard solution onto all samples, matrix spike samples QC samples and blanks.

a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 25ul of the 50ppm 8081A Pesticides QC standard.

5. Immediately add 10.0ml hexane to the vial.
6. Tune sonicator daily or after changing the horn (use 1/8 inch-tapered microtip).

a) Ensure that the probe is not immersed in liquid and that it does not come in contact with anything.
b) Set PULSER to OFF.
c) Set OUTPUT CONTROL to "5" **Never exceed this level with the microtip.**
d) Set POWER SWITCH to ON. The switch will illuminate.
e) Momentarily depress TUNE SWITCH and rotate the tuner clockwise or counterclockwise until a minimum (not maximum) reading (usually <20) is obtained on power monitor.
f) Set OUTPUT CONTROL to "5"
g) Set % DUTY CYCLE to "50"
h) Set TIMER to "120S"
i) Set PULSER to ON position

7. Before each sample, place the probe tip in fresh hexane and energize the power supply for a few seconds to remove any residue.
8. Disrupt the sample with the 1/8 inch tapered microtip ultrasonic probe for 2 minutes at output control setting 5 and with a 50% duty cycle.
9. Loosely pack a disposable Pasteur pipette with 2-3cm glass wool. Filter approximately 4ml of extract through the glass wool into a 4ml vial. *Note final volume in Extraction Log as 10.0ml.
10. Proceed to Vial Labeling and Extract Archival (Sec. X.H)

E. Continuous Liquid-Liquid Extraction (SW-846 Method 3520C)

1. *Pre-rinse all extraction apparatus with $MeCl_2$ before use.
2. Using a 500-mL graduated cylinder, measure 500-mL of sample. Alternatively, a smaller sample volume may be taken and diluted to 500-mL with DI.
3. Spike 20ul of the 25ppm surrogate standard solution into all samples, matrix spike samples QC samples and blanks.
    a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 10ul of the 50ppm 8081A Pesticides QC standard.
4. Check the pH of the sample with wide-range pH paper to ensure a neutral pH between 5 and 9.
5. With stopcock between extractor body and round bottom flask closed add 450-mL methylene chloride directly to the extractor body followed by the 500-mL sample. Rinse the graduated cylinder with 50-mL methylene chloride and add to extractor body.
6. Add several boiling chips to the round bottom flask and secure it to the sidearm of the extractor body. Open the stopcock between the extractor body and the round bottom flask and allow the methylene chloride to pour into the round bottom flask. Add an additional 200-mL DI to the extractor body so that ~300-mL methylene chloride is in the round bottom flask and ~200-mL methylene chloride remains in the bottom of the extractor body.
7. Attach the extraction condenser to the top of the extractor body and run cool water through it. Set the heating mantle so that no methylene chloride escapes through the condenser and extract for 18-24 hours.
8. Allow the extractor to cool. Transfer all remaining methylene chloride extract from the extractor body into the round bottom flask. Decant the solvent extract through a glass funnel containing sodium sulfate and into an 500mL Erlenmeyer flask.
    a) Plug a glass funnel with glass wool and add sodium sulfate until it is 2-3cm above the glass wool plug.
9. Rinse the sodium sulfate with two 30ml portions of methylene chloride.

10. Perform the concentration using the TurboVap Concentration Method (Sec. IX.G)

F. Soxhlet Extraction (SW-846 Method 3540C)

1. *Pre-rinse all extraction apparatus with $MeCl_2$ before use.
2. Blend 10g of the solid sample with 10g anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble.
3. Spike 20ul of the 25ppm surrogate standard solution onto all samples, matrix spike samples QC samples and blanks.
    a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 10ul of the 50ppm 8081A Pesticides QC standard.
4. Place ~250-mL of 1:1 acetone/hexane into a 500-mL round bottom flask containing one or two clean boiling chips and attach it to the bottom of the extractor. Add ~50-mL 1:1 acetone/hexane (enough to cover the sample in the thimble) directly into the extractor.
5. Attach the extraction condenser to the top of the extractor and run cool water through it. Set the heating mantle so that no 1:1 acetone/hexane escapes through the condenser and extract for 16-24 hours at 4-6 cycles/hour.
6. Allow the extractor to cool. Transfer all remaining 1:1 acetone/hexane extract from the extractor into the round bottom flask. Decant the solvent extract through a glass funnel containing sodium sulfate and into an 500mL Erlenmeyer flask.
    a) Plug a glass funnel with glass wool and add sodium sulfate until it is 2-3cm above the glass wool plug.
7. Rinse the sodium sulfate with two 30ml portions of 1:1 acetone/hexane.
8. Perform the concentration using the TurboVap Concentration Method (Sec IX.G)

G. Turbo Vap Concentration Method (nitrogen blowdown technique)

1. *Pre-rinse all extraction apparatus with $MeCl_2$ before use.
2. Transfer a maximum 200-mL of the extract to a 200-mL TurboVap concentrator vial. As the volume in the concentrator vial decreases, additional extract can be added.
3. Place the concentrator vial into the TurboVap II workstation at a water bath temperature of 42 deg C. If the concentrator vials are initially full, start with a low gas pressure and increase the pressure as the volume in the vial decreases. Operate at a maximum gas pressure of 15psi (1.0 bars).
    a) Caution: when the volume of solvent is reduced below 1-mL, analytes may be lost.
4. When the solvent volume reaches ~1.5-mL (at the bottom of the tapered part of the vial) remove the vial from the water bath and add ~10-mL of Hexane. Swirl the concentrator vial until the extract and the hexane are fully mixed, place vials back in the TurboVap II and concentrate to ~1.5-mL.
5. With a 9.0” Pasteur pipette, use the entire sample volume to rinse the lower,

tapered portion of the concentrator vial a couple of times. Transfer the extract to a 4-mL vial.

6. Repeat Step 6, two times with fresh 1-mL portions of hexane.
7. Bring extract up to a final volume of 4-mL.
    a) Proceed to Vial Labeling and Extract Archival (Sec. IX.H).

H. Vial Labeling and Extract Archival

1. All sample extracts are to be archived in 4.0-mL vials in the sample extract refrigerator for a minimum of 40 days.
2. For each sample, a portion of the extract to be analyzed is to be transferred to a 2.0-mL vial lined with a 500uL micro-insert if necessary.
3. All vials are to be labeled with the following:
    a) Method ID (8081A) & Matrix
    b) Sample Reference Number
    c) Extraction Date
    d) Extractionist Initials

I. Sulfur Cleanup with Copper Powder (SW-846 Method 3660B)

1. The recovery of endrin aldehyde is severely reduced by this cleanup method. If sulfur cleanup is to be employed, an untreated portion of the extract must be provided to the analyst for determination.
2. Transfer ~1.0-mL sulfuric acid cleaned extract to a 2mL vial and add copper powder until it reaches ~0.5mL in the vial.
    a) If copper powder does not have a bright shiny appearance it should be treated with dilute nitric acid, rinsed with DI to remove all traces of acid, rinsed with acetone and dried under a stream of nitrogen. This treatment is to ensure that the copper powder is very reactive.
3. Cap the vial and shake vigorously for 1 minute.
4. Allow the phases to separate for at least 10 minutes to ensure that all of the fine copper powder settles out of the extract.
5. Transfer a portion of the extract to a 2mL vial with a 500uL micro-insert and retain the 2mL vial with the copper powder and extract with the archived extracts.

J. Analysis

1. A single-column analysis is performed using a 0.32mm ID Restek Rtx-CLPesticides capillary column (or equivalent).
2. HP G1701BA software is employed to operate the GC/ECD and acquire/store raw data on the 608_ECD computer.
3. GC operating conditions are determined by the analyst and recorded in the following places:
    a) Gas flows are recorded in the instrument maintenance log.
    b) Temperature, pressure and injection programs are all saved as acquisition method AQ-8080. These conditions are printed and filed with the calibration method and chromatograms after a new calibration method

is created.

4. Established operating conditions are used for both calibration and sample analyses.

5. Because of the low concentrations of pesticide standards injected on the GC/ECD, column adsorption may be a problem with the GC has not been used for a day or more. A 10ug (in 4mL hexane) pesticide standard is to be injected prior to beginning the initial calibration or calibration verification in order to prime (or deactivate) the column. This standard should be followed by a hexane blank to ensure there is no carryover.

K. Dilutions

1. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes.

2. Dilutions are made using hexane and must be re-analyzed with all necessary calibration verification requirements being met.

L. Confirmation

1. If analytes are detected in samples at levels near or above the reporting limit, the sample must be quantitatively analyzed on a second, dissimilar column and meet the same RT and calibration criteria as the initial analysis.

2. If the confirmation result is significantly higher (e.g. >40%) and no anomalies are noted, report the higher result and qualify the disparity between results.

3. Confirmation may not be necessary if the composition of the sample matrix is well established by prior analysis.

M. Percent Solids

1. Dry weight, used for soil calculations, is determined by measuring the percent solids of the sample.

2. The BOD/Solids analyst determines percent solids using EPA Method 160.3.

X. Calibration

A. Standards

1. All calibration standards are recorded in the ECD Standards log at the ECD workstation. Standards opened and used in the extraction lab are recorded in the Extraction Lab Standards log.

2. A 10ppm Primary Dilution Standard (PDS) containing all of the single component analytes is prepared by dilution of the purchased pesticide and surrogate mixes with Hexane. The PDS is then used to prepare calibration standards by further dilution with Hexane.

a) A minimum of 5 pesticide calibration standards should be prepared so that the final volume is 4mL.

b) Typical pesticide calibration standards are 0.05ug, 0.1ug, 0.25ug, 0.5ug, 0.75ug and 1.0ug.

3. Multi-component analytes (chlordane and toxaphene) employ a single point calibration near the mid-point of the expected calibration range. Endyne has established a calibration range of 0.2 to 5.0ug of either compound in 4.0mL hexane so a 2.5ug standard of each chlordane and toxaphene is injected after the initial calibration standards for the single component analytes. These standards are used to screen for chlordane and toxaphene based on pattern and peak retention time. If either multi-component compound is recognized in a sample, the following procedure is to be followed for quantitation.

a) Use the 2.5ug standard to estimate the concentration of the target analyte in the sample.

b) Adjust the sample so that the concentration in the extract is ~2.5ug.

c) Inject the 2.5ug calibration standard along with a standard at the quantitation limit (0.5ug) and create a two-point calibration curve using 4-6 of the largest peaks.

d) Inject the adjusted sample and calculate its concentration.

4. If deemed necessary for a specific project, a full calibration 5+ standard calibration may be employed.

B. Initial Calibration

1. An external standard calibration procedure is used for initial calibration.
2. Linear Calibration Using the Response Factor

a) The HP 1701BA Environmental Data Analysis software is used to determine the calibration factor (CF) and relative standard deviation (RSD) for each analyte from a minimum of five calibration standards.

b) If the RSD is less than or equal to 20% over the calibration range the response factor may be used to determine sample concentrations.

c) If the RSD exceeds 20% or at the discretion of the analyst, another calibration method listed below may be used.

3. Linear Calibration Using a Least Squares Regression

a) The HP 1701BA Environmental Data Analysis software is used to perform a linear regression of the instrument response versus the concentration of a minimum of five calibration standards.

b) A weighted least squares regression may be used if replicate multi-point calibrations have been performed. For all other instances an unweighted least squares method should be used.

c) The analyst should not force the line through the origin nor include the origin as an additional calibration point. The intercept is to be calculated from the five data points.

d) The regression calculation will generate a correlation coefficient (r) that is a measure of good fit. When r is greater or equal to 0.990 this calibration method may be used to determine sample concentration.

4. Non-linear Calibration Using a Quadratic Model

a) The HP 1701BA Environmental Data Analysis software is used to

perform a quadratic calibration of the instrument response versus the concentration of a minimum of six calibration standards.

b) The curve may not be forced through the origin nor may the origin be included as a calibration point.

c) Weighting using a quadratic calibration model may significantly improve its accuracy.

d) A coefficient of the determination greater than or equal to 0.990 indicates that the curve may be used to determine sample concentration.

e) The quadratic model is not to be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance.

5. Whichever calibration option is employed, analyte and surrogate concentrations must fall within the calibration range in order for calculations to be valid. Dilutions may be necessary in order to bring analyte concentrations within this range.

6. Individual analytes and surrogates within the method may be calibrated using any of the three methods described above.

7. If initial calibration does not meet acceptance criteria the following steps need to be taken:

a) Check to be sure that there are no errors in the calculations or preparation of calibration standard solutions. If a standard is found to be prepared wrong a new standard should be made and injected.

b) Examine chromatograms for interfering peaks and integrated peak areas. If coelution with contaminant peaks is an issue, instrument conditions may need to be changed to achieve better separation or new calibration standards may need to be made with new reagents.

c) Check instrument performance. If an instrument performance problem is found, correct the problem and re-analyze the initial calibration standards.

d) If no instrument problem is found and reanalysis of initial calibration standards is unsuccessful, new calibration standards must be made.

C. Calibration Verification

1. Before analysis of samples and extracted QA can begin the initial calibration is to be verified by the analysis of a calibration standard. If the calculated concentrations of the analytes in the CV are within +/- 15% of the initial calibration, then the initial calibration is considered still valid.

2. Calibration Verification (CV) standards must also be run at the beginning of each 12-hour analytical shift or after each group of twenty samples (including blanks, LFBs and other QA samples), whichever is more frequent. It is recommended that a CV be run more frequently to minimize the number of repeat injections.

3. CV analyte concentrations must fall within +/- 15% of the initial calibration for the calculated value of preceding samples to be considered accurate. If the CV falls outside of the acceptance limits, all samples back to the last passing CV

must be reanalyzed.

4. A closing CV must also be analyzed at the end of each analytical shift.

5. If initial calibration standards are analyzed at the beginning of a sequence with a valid calibration resulting, the calibration does not need to be verified until the 12-hour analytical shift beginning 12-hours after the first calibration standard was injected or 20 samples have been injected, whichever is less.

6. If calibration verification results fall outside of acceptance limits, a new standard curve must be prepared unless instrument maintenance corrects the problem.

D. Laboratory Performance Check Standard (LPC)

1. Active sites within the analytical system can cause the degradation of 4,4'-DDT into 4,4'-DDE or 4,4'-DDD and Endrin into Endrin ketone or Endrin aldehyde.

2. A LPC containing 1ug each 4,4'-DDT and Endrin in 4.0mL Hexane is to be analyzed for degradation before initial calibration and before each 12-hour analytical shift.

3. Percent breakdown is calculated by dividing the sum of the degradation peak areas by the sum of all peak areas and multiplying by 100. This calculation is to be done separately for Endrin and its degradation byproducts and DDT and its degradation byproducts.

4. Percent breakdown cannot exceed 15% for standards and samples to be analyzed. If breakdown for either DDT or Endrin does exceed 15%, instrument maintenance including changing injection port liners and gold seals and clipping the head of the column, must be performed. A passing LPC must be analyzed before an analysis sequence can begin.

XI. Data Reduction

A. Aqueous calculations

1. Calculated analyte masses in micrograms (ug) are divided by the volume of sample in liters (L) resulting in calculated sample concentrations in ug/L.

2. Concentrations may also need to be adjusted for dilutions by multiplying the final concentration by the dilution factor.

B. Soil calculations

1. Soils are reported out as a dry weight concentration.

2. Cs = Cex/(Ms*(PS/100) where;
   a) Cs = Concentration in the sample (ug/Kg, dry)
   b) Cex = Calculated mass in ug of analyte in the final extract
   c) Ms = Mass of the sample in Kg
   d) PS = Percent solid

3. Concentrations may also need to be adjusted for dilutions by multiplying the final concentration by the dilution factor.

XII. Data Recording

A. Locating the 8081A Chain-of-Custody and extracted samples

1. The Chain-of-Custody for extracted 8081A samples will be found in the bins dedicated to methods of the Organic Department labeled "8081A".
2. Extracted samples are located in the Organics Department refrigerator found in the extraction lab.
3. Enter the individual sample information found on the Extraction Log sheet into the Excel Run-log ("ECD").

B. Target Compounds

1. If analyte concentration is below the reporting limit, report "< (Reporting Limit)"
2. As long as it is within the calibration curve, report that value. Otherwise, dilute the sample and rerun OR add additional standards to the calibration tables. If a target compound is present, then a confirmation process must be done.

C. Unidentified Peaks (UIP)

1. Count the number of non-target peaks equal in size or larger than target peaks near the MRL relative to the extracted LFB
2. Report as UIPs
3. If there are more than 10 UIPs, report as > 10.

XIII. Data Archiving

A. Hard Copies

1. All QC packets from method analyses are to be kept for 5 years.
2. QC packets are separated by method and kept in the filing cabinet near the 608 workstation or in banker boxes (the most recent QC packets are kept in the filing cabinet, space permitting, while all others are kept in folders in labeled banker boxes beneath the workstation).
3. Only the previous and present year QC data are kept on the premises. All other QC data is kept in the Endyne storage facilities.
4. Hard copies of all chromatograms are saved as data packets by order ID with all other Organic data packets.

B. Electronic Backup

1. An electronic backup of all new data files should occur at least once a month.
2. Data is archived on the Unit C computer in the ECD backup folder.
3. Annually, data is backed up on CD-R.

XIV. Instrument Maintenance:

A. Recalibration

1. In addition to recalibration being necessary when calibration verification does not meet acceptance criteria, significant maintenance activities or hardware changes may also require recalibration.
2. Maintenance that should not automatically require recalibration:
   a) Changing septa; compressed gas cylinders; syringes; moisture, hydrocarbon, or oxygen traps; split seals; column fittings; inlet liners.
   b) Other procedures include breaking off or changing a guard column or cleaning the inlet.
   c) Whenever such procedures are performed, the analyst must demonstrate that the results for a calibration verification standard meet the acceptance criteria.
3. Maintenance that automatically requires recalibration:
   a) Changing, replacing, or reversing the column; and replacing the injection port.
   b) Changing or replacing the column will also require that the retention time windows be redetermined.

B. Maintenance Log

1. In the Instrument Log, enter any problems that have arisen with any methods and how the problem was solved. Also log in any changes made to the instrument (i.e. gas flow changes, column changes, injection port changes, etc.).
2. Each entry must be accompanied by the Date and Analyst's Initials

C. Standards Log

1. Whenever any standard is opened or made up, enter the date, analyst's initials, source of standard, type and concentration, and detailed steps as to how the standards were prepared.

D. Maintenance

1. General
   a) The Merlin Micro Seal is good for more than 2000 injections. However, if the traditional septa are used, they must be changed every 30 or so runs.
      (1) Occasional inspection of the microseal for coring or cracking is recommended.
   b) Replace injection port liner every 100-150 injections; whenever standard degradation is evident
   c) Replace injection port orange O-Rings if dried and cracking
   d) Replace column ferrules when cutting the column
2. Injection Port Liner Cleaning
   a) Rinse with $MeCl_2$

b) Rinse with HNO_3
c) Rinse with HCl
d) Rinse with DI
e) Rinse with MeOH
f) Dry with N_2
g) Rinse for 30 seconds with silanating agent
h) Rinse with Toluene
i) Rinse with MeOH
j) Dry with Nitrogen

3. Changing a Column
   a) Lower all GC temperatures (oven, injector, and detector) to < 70°C
   b) Remove the detector end of the column first
   c) Immediately cap the ECD with a plugged ferrule nut
   d) Plug the detector end of the column with and injection port septa
   e) Remove the injector end of the column and plug it into the septa from Step "d"
   f) Remove the old column from the oven and install the new column by reversing Steps "a" – "e"

XV. Trouble Shooting:

A. No Signal

1. Make sure that the column is intact and has the proper flows
2. Make sure that the proper detector is selected in the method that is loaded

B. Noisy / High Baseline

1. Using an air duster, check for leaks into the system by spraying at system joints from injector to detector. If a leak is present, there will be sharp spikes in the signal.
2. Double check column insertion distances. The column on the detector end should extend about 6.5 cm from the ferrule, and the column on the injector end about 2mm.
   a) The best way to achieve these distances is by installing the column with the new ferrules, but leaving them loose enough to push/pull on the column. Then, push the column all the way in and then back it off about 2mm. Then tighten the ferrule.
3. Cap detector and bake for 3-6 hours. It may be necessary to raise the temperature to 350 or 400°C. DO NOT LEAVE AT 400°C OVERNIGHT. After this, the signal should fall and remain stable.
4. If after baking the signal is still too high and there are no leaks, then it is probably time to remove the detector and ship it out to be professionally cleaned thermally and chemically.
5. If after baking, the signal seems acceptable, then re-install the column. If the signal goes up and remains high, then there may be contamination on the column. This contamination should be removable by baking the column (removed from the detector) at 310 - 320°C for 3-4 hours.

C. High or Low Column Flows / Head Pressure Not Adjustable

1. Split vent lines may be contaminated with sample waste. To resolve this problem, rinse the purge lines with MeOH and blow dry.
2. The column may be plugged. Check the detector and injector ends of the column (using XIII:3C) for blockage

D. No Head Pressure

1. Make sure the injector column fittings are tight.
2. Make sure the "Total Flow" valve is open.

E. Reduction in Response / Coelution of Certain Analytes

1. There may be active sites in the injector / column. Try changing the injection port liner, gold plated seal, septa, or removing 1-2 loops of column.
2. Check column flow. If flow is too high or too low, coelution and/or tailing may occur.

XVI. Method Modifications:

1. Sec. IX.D. Method 3550B calls for the solvent to be decanted through filter paper after sonication. Endyne filters through muffled sodium sulfate and glass wool to remove particulate and further dry the extract.

XVII. Bibliography/References:

Method 8081A, Organochlorine pesticides by Gas Chromatography, Revision 1. EPA SW-846 NTIS, Dec. 1996.

Method 8000b Determinative Chromatic Separations, EPA SW-846 NTIS, CD-ROM Version 2, Dec. 1997

GC Trouble Shooting Guide, 3rd edition, Phenomonex, August 1999

HP 5890A Series II Reference Manual, fifth edition, Hewlett-Packard, Sept. 1991 Publication # 05890-90270

HP 7673 Automatic Sampler, forth edition, Hewlett-Packard, June 1992 Publication # 07673-90185

Operating Hints for Split/Splitless Injectors, Restek, 1992. Bellefonte, PA.

A Guide When Injecting Dirty Samples, Restek, 1994. Bellefonte, PA.

XVIII. Revision History:

A. from revision #7 revision #8

1. Added Bibliography/Reference section; Added Revision History section; Added sign off sheet.
2. Edited typos; formatted tab sets and spacing; updated table of contents
3. Added laboratory safety requirements.

B. Revision #9(Matthew White)

1. Complete revision of extractions and cleanups to include all methods used in the lab. Also added sonicator calibration instructions.
2. Clarified calibration section to include all linear and non-linear calibration techniques, calibration verification and LPC criteria.
3. Revised summary and introduction as well as reporting limits and QA sections.
4. Expanded Quality Control section to include more detailed information about the acceptability of QA/QC samples and surrogates as well as protocol for out of control QA/QC.

I acknowledge that I have read this Standard Operating Procedure and agree to adhere to the Safety and Quality Control policies outlined within.

Name	Date

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POLYCHLORINATED BIPHENYLS (PCBs)
Method EPA 8082

Standard Operating Procedure

REVISED: February 8, 2007

Revision #: 10

Technical Director: _____Date :_____
Harry B. Locker Ph.D.

QA Officer: _____Date :_____
Mark L. Fausel

Document Control Copy on Buff colored paper. Any other color is not official version

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I. Scope and Application

A. Overview

Method 8082 is used to determine the concentrations of various polycholinated biphenyls (PCBs) in extracts from solid and liquid matrices. Samples are extracted by appropriate EPA 3500 series methods, concentrated to 4.0-mL using a Zymark TurboVap II workstation and cleaned using EPA 3600series methods. The extract is analyzed by GC/ECD using Restek Rtx-CLPesticides (or equivalent) capillary column. The compounds listed below are determined by a single column analysis system and confirmed on a Restek Rtx-CLPesticides II (or equivalent) capillary column.

1. Compounds and Reporting Limits

Compound	Water	Soil
Aroclor 1016	< 0.5 µg/L	< 10.0µg/kg
Aroclor 1221	< 0.5 µg/L	< 10.0µg/kg
Aroclor 1232	< 0.5 µg/L	< 10.0µg/kg
Aroclor 1242	< 0.5 µg/L	< 10.0µg/kg
Aroclor 1248	< 0.5 µg/L	< 10.0µg/kg
Aroclor 1254	< 0.5 µg/L	< 10.0µg/kg
Aroclor 1260	< 0.5 µg/L	< 10.0µg/kg
Aroclor 1268	< 0.5 µg/L	< 10.0µg/kg

2. The analysis of organochlorine pesticides should be undertaken using Method 8081A, which included specific cleanup and quantitation procedures designed for pesticide analysis. If both the analysis of organochlorine pesticides and PCBs are requested for a particular sample, split the extract prior to any cleanup steps and process part of the extract for organochlorine pesticide analysis using Method 8081A and the other portion for PCB analysis using Method 8082.

3. Method 8082 is used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors or as individual PCB congeners in extracts from solid and aqueous matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). When compared to packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The target compounds listed above may be determined by either a single- or dual-column system.

4. Matrix specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.).

5. When samples contain more than one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation.

6. Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique.

7. This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

B. Summary of Method

1. A measured volume or weight of sample (approximately 1-L for liquids and 2g – 30g for solids) is extracted using the appropriate matrix-specific sample extraction technique.
   a) Liquid samples are extracted at a neutral pH (pH 5-9) with methylene chloride using Method 3510 (separatory funnel) or Method 3520 (continuous liquid-liquid extractor).
   b) Solid samples are extracted with 1:1 acetone/methylene chloride using Method 3550 (ultrasonic extraction) or with 1:1 acetone/hexane using Method 3540 (soxhlet extraction).
   c) Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for there analytes. This clean up technique will remove many single component organochlorine and organophosphorus pesticides. Therefore, Method 8082 is not applicable to the analysis of those compounds. Method 8081A should be used instead.
   d) In addition, a sulfur cleanup using copper powder (Method 3660) may be used if sulfur is known to be present in the extract.
2. After cleanup, the extract is analyzed by injecting a 2-uL sample into a gas chromatograph with a fused silica capillary column and electron capture detector (GC/ECD).

II. Safety

1. This method involves the handling of the chemicals outlined below. Each of these chemicals may have specific associated hazards. Analysts must read and become familiar with all elements of each chemical's MSDS. Make special awareness of the symptoms of exposure and potential routes of entry. Analysts must notify the Safety Officer immediately if any symptoms of exposure are suspected.
2. PPE must be worn at all times in designated work areas. All applicable

engineering controls, such as fume hoods or splash shields, must be used when performing analyses. To ensure the safety of all laboratory personnel; work areas must be kept neat and organized, equipment and bench-tops must be cleaned and sterilized, if necessary, at the completion of each task, all containers must be properly labeled as to contents and hazard ratings and spills cleaned immediately and completely.

3. Lab coats, Nitrile Gloves, and Protective Eyeglasses are to be worn at all times during analysis by this method and when handling any laboratory sample.
4. A thorough MSDS review of Methylene Chloride, Sodium Sulfate, Methanol, Hexane and all laboratory calibration standards should be performed prior to analysis by this method.
5. All standards should be made in fume hood away from heat sources.
6. This method uses reagents that may cause anemia, eye/liver/kidney/central nervous system disorders, reproductive disorders and cancer upon exposure. Stock standards of these materials should be handled in fume hoods. Toxic gas respirators should be worn when handling concentrated standards and samples.

III. Waste Management:

A. Solvent Waste:

All solvent waste must be disposed of in the solvent waste satellite container, then placed in the larger container located in the hazardous waste room. Note: Dumping of solvent waste in the Hazardous waste room can ONLY be done by a Technician certified to do so.

B. Water/Soil Waste:

Solid and aqueous material must sit in a vented area until all the solvent has been evaporated. Alternatively, it may be collected in a carboy and bubbled with the exhaust of the pump in the extraction lab hood. The water can be flushed down the drain with copious amount of tap water. Soil waste is stored in a satellite container and disposed of in the solid waste container in the hazardous waste room.

C. Acid Waste:

Acid waste should be contained separate from all other waste streams and disposed of in the proper waste container in the hazardous waste room by a technician who is trained to do so.

IV. Sample Collection, Preservation, and Storage:

A. Sample Collection and Preservation

1. Collect water samples in 1L amber glass and preserve with Sodium Thiosulfate.
2. Adjust the pH to 5.0 - 9.0, using 1:1 H_2SO_4 or 10N NaOH as needed, if the sample is not going to be extracted within 72 hours.
    a) Record the volumes added on the Chain-of-Custody and store the preserved sample in a 4^oC refrigerator.
3. Collect solids in 250mL amber glass, and refrigerate at 4^oC.

B. Storage

1. All samples are stored in the Extraction Lab refrigerator at 4^oC.
2. All extracts are stored in the Sample Extract refrigerator in the extraction lab at 4^oC.
3. Standards are stored in the Extraction Lab and Organics Lab Standard refrigerators at 4^oC.

C. Holding Times

1. Extraction
    a) Waters: 7 days from sampling
    b) Solids: 14 days from sampling
2. Analysis
    a) 40 days from extraction

V. Equipment and Supplies

A. Analysis

1. Desktop computer running HP Chemstation 1701 BA
2. Hewlett-Packard 5890 GC
3. Hewlett-Packard 19233 Electron Capture Detector
4. Hewlett-Packard 7673 Autosampler
5. Rtx-CLPesticides (or equivalent) column: 30m, 0.32mm Id, 0.5um film – primary analysis.
6. Rtx-CLPesticides II (or equivalent) column: 30m, 0.32mm Id, 0.25um film – confirmation analysis.
7. Merlin microseal – septa
8. Cyclosplitter injection port liner (or equivalent)
9. J&W Electronic Flow Meter
10. Vent Carbon Trap
11. Ultra-High Purity N_2 gas supply

B. Extraction

1. 375 watt Ultrasonic Disrupter (*Tekmar*)
   a) ¾" horn (low concentration)
   b) 1/8" tapered microtip attached to a ½" horn (high concentration)
2. 250mL Beakers
3. 500mL Erlenmeyer Flasks
4. Glass funnels
5. Various sized syringes
6. 2 Liter *Teflon* separatory funnels
7. 1 Liter graduated cylinder
8. 9" Glass Pasteur pipettes
9. 2mL and 4mL vials, *Teflon* septa caps, and micro inserts
10. Metal spatulas
11. *Zymark* TurboVap II concentrator
12. *Zymark* TurboVap II concentration vials
13. Soxhlet extractors with 500mL round bottom flasks
14. Continuous liquid-liquid extractor with 500mL round bottom flask
15. Heating mantle – Rheostat controlled
16. Boiling chips – solvent rinsed

VI. Reagents and Standards:

A. Reagents Log

1. All reagents are recorded in the Reagents Log when opened. The date reagents are entered into the Reagents Log is then used to trace reagents on the Extraction Log.

B. Reagents

1. Reagent grade chemicals that conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.
2. Hexane – Fisher Optima
3. Acetone – Fisher Optima
4. Methylene chloride – GC Resolv
5. Methanol
6. Anhydrous granular sodium sulfate – Certified ACS
   a) Purify by heating to 400 ^{o}C for 4 hours in a shallow tray.
   b) The sodium sulfate log is kept by the analyst who is responsible for baking the sodium sulfate in the muffle furnace. Reagent information as well as baking information (time, temperature…) is recorded here.
7. De-ionized water – produced by Endyne, Inc

8. Pool sand – Methylene chloride rinsed
9. Glass wool – Silanated
10. Concentrated sulfuric acid
11. 10 N NaOH
12. Nitric acid – dilute
13. Copper powder – remove oxides by treating with dilute nitric acid, rinse with DI to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen.

C. Standards

1. Store standards at 4 ^{o}C in PTFE-sealed containers in the standards refrigerator in the extraction lab.
2. Ensure that all purchased standards are in hexane, acetone or solvents compatible with hexane.
3. Standards purchased for QCS samples should come from a separate supplier than calibration standards – when possible.
4. AccuStandard C-2xxS-H (100ppm Aroclor Mix) for Aroclors 1221, 1232, 1242, 1248, 1254, 1268 or equivalent – 2 lots, one for calibration and one for QC.
5. Accustandard C-2xxS-H-10X (1000ppm Aroclor Mix) for Aroclors 1016 and 1260 or equivalent – 2 lots, one for calibration and one for QC
6. AccuStandard CLP-032-R (200ppm decachlorobiphenyl and tetrachloro-m-xylene) or equivalent
7. PCB QC mix (1016/1260, 100ppm):
   a. Prepare by spiking 800ul of Methanol with 100uL of the 1000ppm Aroclor 1016 mix and 100ul of the 1000ppm Aroclor 1260 mix (final volume of 1.0ml).

VII. Interferences:

A. Sources

1. Contaminated solvents, reagents, or sample processing hardware.
2. Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.
3. Compounds extracted from the sample matrix to which the detector will respond.

B. Prevention and Solutions

1. Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. These materials can also be removed through the use of Method 3665.
2. Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed

by detergent washing with hot water, and rinses with tap water and DI water. Drain the glassware and dry it in an oven at 130 ^{o}C for several hours or rinse with MeOH and drain. Volumetric glassware should not be subjected to heating. Store dry glassware in a clean environment.

3. Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of Method 3660.

VIII. Quality Control:

A. Extraction

1. The following should be extracted at least 1 per set or 1 per 20 samples, whichever is more frequent:
2. Laboratory Reagent Blank (LRB)
   a) 1.0L DI H_2O or 30.0g pool sand spiked with 20uL of 50ppm TCMX & DCB
3. Laboratory Fortified Blank (LFB)/Quality Control Sample (QCS)
   a) 1.0L DI H_2O or 30.0g pool sand spiked with 20uL of 50ppm TCMX & DCB, and 30uL of 100ppm 1016/1260 QC mix.
   b) The stock standard PCB mix should come from a source independent from the calibration stock standard.
4. Laboratory Duplicate (LD)
   a) A sample aliquot taken in addition to the first, spiked with 20uL of 50ppm TCMX & DCB, extracted, and analyzed with an identical procedure to the first in order to measure the precision associated with this laboratory procedure.
5. Laboratory Fortified Sample Matrix (LFM)
   a) A sample aliquot taken in addition to the first, and spiked the same as an LFB to determine whether the sample matrix contributes bias to the analytical results.

B. Method Blanks/LRB

1. When samples that are extracted together are analyzed on separate instruments or on separate analytical shifts, the method blank must be analyzed on at least one of those instruments.
2. The method blank may be analyzed immediately after the calibration verification standard, to ensure that there is no carryover from the standard, or at another point in the analytical shift.
3. When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.
4. LRBs should have target compounds:
   a) Less than the reporting limit for the analyte;
   b) Less than 5% of the regulatory limit associated with an analyte;
   c) Less than 5% of the sample result for the same analyte, whichever is

greater.

5. If the method blank results do not meet the acceptance criteria above, then the laboratory should take corrective action to locate and reduce the source of the contamination and to re-extract and reanalyze any samples associated with the contaminated method blank.
6. If method blank does not meet acceptance criteria and samples are past holding times, reported data will be qualified "B" indicating that blank contamination was observed at levels affecting analytical results.
7. Blank subtraction is not permitted.
8. Method blanks and/or solvent blanks may be used to check for contamination by carryover from a high-concentration sample into subsequent samples.
9. When analysis of such blanks is not possible the analyst should review the result for at least the next two samples after the high-concentration sample.
   a) If the analytes in the high-concentration sample are not present in subsequent samples, the lack of carryover has been demonstrated.
   b) If there is evidence that carryover may have occurred, then the affected samples should be reanalyzed.

C. LFB/QCS sample

1. The LFB consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. It is fortified at the same concentration as the matrix spike.
2. Recovery calculations are as follows: %Recovery = ((Cs – Cu)/ Cn)*100 where:
   a) Cs = Measured concentration of the spiked sample aliquot
   b) Cu = Measured concentration of the unspiked sample aliquot (use 0 for the LFB)
3. Cn = Nominal (theoretical) concentration increase that results from spiking the sample or the nominal concentration of the spiked aliquot (for the LFB should fall within established laboratory control limits. In the absence of control limits, 70-130% may be used.
4. If the LFB falls outside of acceptance limits, the laboratory should take the following corrective action:
   a) Check to be sure that there are no errors in the calculations or standard solutions. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.
   b) Check instrument performance. If an instrument performance problem is found, correct the problem and re-analyze the extracts as well as all associated sample extracts.
   c) If no instrument problem is found, the analytical batch should be re-extracted and re-analyzed if still within holding time.
   d) If upon re-analysis (of initial extract or re-extracted extract), the recovery is again not within limits, affected analytes should be qualified "QA" indicating that associated QA/QC did not meet laboratory guidelines.

D. Matrix Spike (MS)/LFM sample

1. A duplicate field sample is fortified with a known amount of analyte in order to document the effect of the matrix on method performance.
2. Matrix spikes are fortified with the same amount of analyte as the LFB/QCS.
3. Matrix Spike recoveries are not calculated if the fortified concentration is <20% of the background concentration of the sample.
4. Recoveries for Matrix Spike samples are calculated in the same manner as the LFB/QCS and must meet established control limits.
5. In the absence of control limits, 70-130% may be used.
6. When the results of a matrix spike analysis indicates a potential problem due to the sample matrix itself (a positive of negative bias) the LFB/QCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
7. If it is determined that the matrix is biasing quantitation, affected analytes are to be qualified "M+" or "M-" indicating a positive or negative bias due to the sample matrix.
8. If both the LFB and MS fall outside of acceptance limits, follow the corrective action outlined for the LFB/QCS (sec. VII.C.4).

E. Duplicate/LD

1. A duplicate sample is analyzed along side the sample in order to measure the precision associated with this method.
2. Precision is an estimate of the relative percent difference (RPD) of the concentrations for the analysis of the unspiked samples.
3. RPD = ((C1-C2)/((C1+C2)/2))*100 where:
    a) C1 = measured concentration of the first sample aliquot
    b) C2 = measured concentration of the second sample aliquot
4. Acceptance limits for precision are 70-130% RPD
5. Duplicates that fall outside acceptance criteria should be qualified as "DS," indicating that there were disparate results between laboratory duplicate analyses.

F. Surrogates

1. In addition to the fortification of all QA/QC samples with 20uL of 50ppm TCMX &DCB surrogate mixture, all field samples must also be fortified and evaluated versus in-house acceptance criteria.
2. Surrogate recoveries is calculated as: (Recovered Surrogate Concentration / Surrogate Target Concentration) * 100
3. Surrogate recoveries must fall within established acceptance limits. If surrogate recoveries are outside of acceptance limits the following steps should be taken.
    a) Check to be sure that there are no errors in the calculations or surrogate standard solutions. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.

b) Check instrument performance. If an instrument performance problem is found, correct the problem and re-analyze the extract.

c) Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analysis should be provided to the data user.

d) If no instrument problem is found, the sample should be re-extracted and re-analyzed if still within holding time.

e) If upon re-analysis (of initial extract or re-extracted extract), the recovery is again not within limits, Surrogate value reported to the client must be qualified as "NR" indicating that the surrogate falls outside laboratory QA/QC parameters.

G. Demonstration of Capability (DOC)

1. Prior to extracting samples for clientele, a new extractionist must extract 4 LFB samples at the standard QC concentration. These LFB must be analyzed as other laboratory samples and then processed according to the following acceptance criteria:

a) Calculate the average in ug/L and the standard deviation for each parameter using the four results

b) The average % Recovery must fall within established control limits for LFBs, and the percent standard deviation must be less than 20%.

H. Method Detection Limits (MDL)/Reporting Limits

1. An MDL study should be performed once a year.

a) Seven low-level QCs are extracted. To determine the MDL for each analyte and surrogate, the standard deviation of the seven values is multiplied by 3.14.

b) The target concentration of the low-level QCs should be 2.5-5 times the calculated MDL value.

c) Method Detection Limit should be no greater than one-fifth the Reporting Limit.

2. The reporting limit is based on achieving the requested detection limits of our clients as well as being below known environmental action limits. The laboratory takes into account known interferences, such as contamination peaks. It should be no less than 5 times the MDL.

3. For established reporting limits refer to Section I.

I. Limits, Tables, and Charts

1. Tables of surrogate and analyte recoveries from LFBs and LFMs are maintained, and updated annually. Surrogate data is established from field samples and LFB and LFM data come from LFBs and Matrix Spike samples.
2. Limits are based on the previous chronological year's data, unless significant modification has been made to the methodology in which case limits are established after the first twenty samples have been analyzed.
   a) Control Limits: parameter average +/- 3(Standard Deviation)
   b) Warning Limits: parameter average +/- 2(Standard Deviation)
   c) Control Charts must be separated by matrix and extraction procedure (Ex: Soil by Sonication, Soil by Soxhlet, Soil by Continuous)
   d) Up to date Control Charts are kept in the 608 analyst's workspace.

J. Retention Time Window

1. Retention Time Windows (RTW) are used to determine whether or not a target analyte is present in a sample.
   a) Establish a RTW by first making three (3) injections of calibration standard over a 72-hour period.
   b) Record the RT's for each component to the third decimal place.
   c) Calculate the mean and standard deviation for each component.
   d) For multi-peaked compounds, choose 4-6 of the biggest peaks
   e) RTW Width: +/- 3(STDEV) of the absolute RT average OR 0.03, whichever is greater.
   f) The experience of the analyst should weigh heavily in the interpretation of chromatograms.
2. On subsequent analyses of samples, establish RT's from the first calibration line check.
3. For samples run with the initial calibration, the middle line point is used as the RT reference.
4. RTW must be established for each column used to analyzed samples.

K. Qualifying Data

1. Any deviations from the above outlined quality control as well as any sample container, preservation or holding time discrepancies, outlined in Section IV, are noted on the final report.

IX. Procedures

A. Extraction Log

1. An 8080/608 Extraction Log is to be filled out for every extraction batch and kept in the extraction log binder in the extraction lab. Copies of the extraction log are to be stapled to the Login Reports and given to the analyst when extractions have been completed.

B. Separatory Funnel Liquid-Liquid Extraction (SW-846 Method 3510C)

1. *Pre-rinse all extraction apparatus with $MeCl_2$ before use.
2. Using a 1-L graduated cylinder, measure 1-L of sample. Alternatively, if the entire contents of the sample bottle are to be extracted, mark the level of the sample on the outside of the bottle. If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1-L with DI.
    a) 1-L of DI H_2O is to be used for the blank and QC sample.
3. Spike 20ul of the 50ppm surrogate spiking solution into each sample (including blanks, QC samples and matrix spike samples) in the graduated cylinder (or sample bottle) and mix well.
    a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 30ul of the 100ppm PCB 1016/1260 QC standard.
4. Check the pH of the sample with wide-range pH paper to ensure a neutral pH between 5 and 9.
5. Quantitatively transfer the sample from the graduated cylinder (or sample bottle) to the 2-L separatory funnel. Use 60mL of methylene chloride to rinse the cylinder (or bottle) and transfer this rinse solvent to the separatory funnel.
    a) If the sample was transferred directly from the sample bottle, refill to the mark made in Sec. X.B.2 with water and then measure the volume of sample that was in the bottle.
6. Seal and shake the separatory funnel vigorously for 1- 2 minutes with periodic venting to release excess pressure. The automatic shaker may be employed on the high setting for 2 minutes after the separatory funnel has been shaken by hand and vented periodically for 30 seconds.
    a) Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. The separatory funnel should be vented into a hood to avoid exposure of the analyst to solvent vapors.
7. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques.
    a) The optimum technique depends on the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.
8. Decant the solvent extract through a glass funnel containing sodium sulfate and into a 500mL Erlenmeyer flask.
    a) Plug a glass funnel with glass wool and add sodium sulfate until it is 2-3cm above the glass wool plug.
9. Repeat the extraction (steps 5-8) two more times adding fresh portions of methylene chloride directly to the separatory funnel. On the final extraction, rinse the sodium sulfate with two 30ml portions of methylene chloride.
10. Perform the concentration using the TurboVap Concentration Method (Sec. IX.H)

C. Ultrasonic Extraction (SW-846 Method 3550B)-Low Concentration, <20mg/kg

1. The following steps should be performed rapidly to avoid the loss of the more volatile extractable compounds.
2. *Pre-rinse all extraction apparatus with methylene chloride before use.
3. Weigh approximately 30g of sample into a 500ml beaker. Record the weight to the nearest 0.01g. 30g of methylene chloride rinsed pool sand are to be used for the blank and QC sample.
    a) Samples must be well mixed prior to weighing. Dump entire contents of sample into a tared beaker, remove any foreign objects (sticks, leaves and rocks) and thoroughly mix the sample with a spatula. Replace sample into original sample container leaving 30g in the beaker to be extracted.
4. Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture must be mixed with 60g of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate may be added.
    a) After addition of sodium sulfate, the sample should be free flowing.
5. Spike 20ul of the 50ppm surrogate standard solution onto all samples, matrix spike samples QC samples and blanks.
    a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 30ul of the 100ppm 8082 PCB 1016/1260 QC standard.
6. Immediately add 100mL of 1:1 acetone/methylene chloride.
7. Tune sonicator daily or after changing the horn (use ¾ inch horn).
    a) Ensure that the probe is not immersed in liquid and that it does not come in contact with anything.
    b) Set PULSER to OFF.
    c) Set OUTPUT CONTROL to “10”
    d) Set POWER SWITCH to ON. The switch will illuminate.
    e) Momentarily depress TUNE SWITCH and rotate the tuner clockwise or counterclockwise until a minimum (not maximum) reading (usually <20) is obtained on power monitor.
    f) Set OUTPUT CONTROL to “10”
    g) Set % DUTY CYCLE to “50”
    h) Set TIMER to “180S”
    i) Set PULSER to ON position
8. Before each sample, place the probe tip in fresh 1:1 acetone/methylene chloride and energize the power supply for a few seconds to remove any residue.
9. Place the bottom surface of the tip of the ¾ inch disrupter horn about ½ inch below the surface of the solvent, but above the sediment layer. If the horn comes in contact with the sediment output is reduced.
10. Press START on sonicator and extract ultrasonically for 3 minutes, with output control knob set to 10 (full power) at 50% duty cycle. Do not use microtip probe.
    a) *It is highly advisable to wear earplugs during the sonication process to prevent possible damage to one’s hearing.
11. Decant the solvent extract through a glass funnel containing sodium sulfate

and into a 500mL Erlenmeyer flask.

a) Plug a glass funnel with glass wool and add sodium sulfate until it is 2-3cm above the glass wool plug.

12. Repeat the extraction two or more times with two additional 100mL portions of 1:1 acetone/methylene chloride. Decant off the solvent after each ultrasonic extraction. On the final ultrasonic extraction, pour the entire sample onto the sodium sulfate and rinse with two 50ml portions of 1:1 acetone/methylene chloride.

13. Perform the concentration using the TurboVap Concentration Method (Sec. IX.H)

D. Ultrasonic Extraction (SW-846 Method 3550B)-High Concentration, >20mg/kg

1. The following steps should be performed rapidly to avoid the loss of the more volatile extractable compounds.
2. Transfer approximately 2g (record weight to the nearest 0.01g) of well-mixed sample to a 40mL VOA vial. Wipe the mouth of the vial with a Kimwipe to remove any sample material. Record the exact weight of the sample taken.
3. Add 2g anhydrous sodium sulfate to the sample in the 40mL vial and mix well.
4. Spike 50ul of the 50ppm surrogate standard solution onto all samples, matrix spike samples QC samples and blanks.
   a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 75ul of the 100ppm 8082 PCB 1016/1260 QC standard.
5. Immediately add 10.0ml hexane to the vial.
6. Tune sonicator daily or after changing the horn (use 1/8 inch-tapered microtip).
   a) Ensure that the probe is not immersed in liquid and that it does not come in contact with anything.
   b) Set PULSER to OFF.
   c) Set OUTPUT CONTROL to “5” **Never exceed this level with the microtip.**
   d) Set POWER SWITCH to ON. The switch will illuminate.
   e) Momentarily depress TUNE SWITCH and rotate the tuner clockwise or counterclockwise until a minimum (not maximum) reading (usually <20) is obtained on power monitor.
   f) Set OUTPUT CONTROL to “5”
   g) Set % DUTY CYCLE to “50”
   h) Set TIMER to “120S”
   i) Set PULSER to ON position
7. Before each sample, place the probe tip in fresh hexane and energize the power supply for a few seconds to remove any residue.
8. Disrupt the sample with the 1/8 inch tapered microtip ultrasonic probe for 2 minutes at output control setting 5 and with a 50% duty cycle.
9. Loosely pack a disposable Pasteur pipette with 2-3cm glass wool. Filter

approximately 4ml of extract through the glass wool into a 4ml vial. *Note final volume in Extraction Log as 10.0ml.

10. Proceed to Vial Labeling and Extract Archival (Sec. IX.I)

E. Continuous Liquid-Liquid Extraction (SW-846 Method 3520C)

1. *Pre-rinse all extraction apparatus with $MeCl_2$ before use.
2. Using a 500-mL graduated cylinder, measure 500-mL of sample. Alternatively, a smaller sample volume may be taken and diluted to 500-mL with DI.
3. Spike 20ul of the 50ppm surrogate standard solution into all samples, matrix spike samples QC samples and blanks.
    a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 30ul of the 100ppm 8082 PCB 1016/1260 QC standard.
4. Check the pH of the sample with wide-range pH paper to ensure a neutral pH between 5 and 9.
5. With stopcock between extractor body and round bottom flask closed add 450-mL methylene chloride directly to the extractor body followed by the 500-mL sample. Rinse the graduated cylinder with 50-mL methylene chloride and add to extractor body.
6. Add several boiling chips to the round bottom flask and secure it to the sidearm of the extractor body. Open the stopcock between the extractor body and the round bottom flask and allow the methylene chloride to pour into the round bottom flask. Add an additional 200-mL DI to the extractor body so that ~300-mL methylene chloride is in the round bottom flask and ~200-mL methylene chloride remains in the bottom of the extractor body.
7. Attach the extraction condenser to the top of the extractor body and run cool water through it. Set the heating mantle so that no methylene chloride escapes through the condenser and extract for 18-24 hours.
8. Allow the extractor to cool. Transfer all remaining methylene chloride extract from the extractor body into the round bottom flask. Decant the solvent extract through a glass funnel containing sodium sulfate and into an 500mL Erlenmeyer flask.
    a) Plug a glass funnel with glass wool and add sodium sulfate until it is 2-3cm above the glass wool plug.
9. Rinse the sodium sulfate with two 30ml portions of methylene chloride.
10. Perform the concentration using the TurboVap Concentration Method (Sec. IX.H)

F. Soxhlet Extraction (SW-846 Method 3540C)

1. *Pre-rinse all extraction apparatus with $MeCl_2$ before use.
2. Blend 10g of the solid sample with 10g anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the of the extraction period. A glass wool plug above and below the sample in

the Soxhlet extractor is an acceptable alternative for the thimble.

3. Spike 20ul of the 50ppm surrogate standard solution onto all samples, matrix spike samples QC samples and blanks.
    a) For the sample in each batch selected for use as a matrix spike sample and the QC sample, spike 30ul of the 100ppm 8082 PCB 1016/1260 QC standard.
4. Place ~250-mL of 1:1 acetone/hexane into a 500-mL round bottom flask containing one or two clean boiling chips and attach it to the bottom of the extractor. Add ~50-mL 1:1 acetone/hexane (enough to cover the sample in the thimble) directly into the extractor.
5. Attach the extraction condenser to the top of the extractor and run cool water through it. Set the heating mantle so that no 1:1 acetone/hexane escapes through the condenser and extract for 16-24 hours at 4-6 cycles/hour.
6. Allow the extractor to cool. Transfer all remaining 1:1 acetone/hexane extract from the extractor into the round bottom flask. Decant the solvent extract through a glass funnel containing sodium sulfate and into a 500mL Erlenmeyer flask.
    a) Plug a glass funnel with glass wool and add sodium sulfate until it is 2-3cm above the glass wool plug.
7. Rinse the sodium sulfate with two 30ml portions of 1:1 acetone/hexane.
8. Perform the concentration using the TurboVap Concentration Method (Sec IX.H)

G. PCB in Oil sample preparation

1. Add approximately 0.4g of sample to a tared 4.0ml vial. Record the exact mass of sample on the Extraction Log.
2. For LFB and LRB samples, use PCB-free transformer oil.
3. Spike all samples, LRB, LFB, LD and LFM with 20ul of 50ppm 8082 Surrogate mixture.
4. Spike LFB and LFM samples with 30ul of 100ppm 1016/1260 QC mixture.
5. Add ~3.6ml of hexane to the vial for a final volume of 4.0ml
6. Proceed to Section J (Sulfuric Acid cleanup).

H. Turbo Vap Concentration Method (nitrogen blowdown technique)

1. *Pre-rinse all extraction apparatus with $MeCl_2$ before use.
2. Transfer a maximum 200-mL of the extract to a 200-mL TurboVap concentrator vial. As the volume in the concentrator vial decreases, additional extract can be added.
3. Place the concentrator vial into the TurboVap II workstation at a water bath temperature of 42°C. If the concentrator vials are initially full, start with a low gas pressure and increase the pressure as the volume in the vial decreases. Operate at a maximum gas pressure of 15psi (1.0 bars).
    a) Caution: when the volume of solvent is reduced below 1-mL, analytes may be lost.
4. When the solvent volume reaches ~1.5-mL (at the bottom of the tapered part

of the vial) remove the vial from the water bath and add ~10-mL of Hexane. Swirl the concentrator vial until the extract and the hexane are fully mixed, place vials back in the TurboVap II and concentrate to ~1.5-mL.

5. With a 9.0" Pasteur pipette, use the entire sample volume to rinse the lower, tapered portion of the concentrator vial a couple of times. Transfer the extract to a 4-mL vial.
6. Repeat Step 5, two times with fresh 1-mL portions of hexane.
7. Bring extract up to a final volume of 4-mL.
    a) Proceed to Vial Labeling and Extract Archival (Sec. IX.I).

I. Vial Labeling and Extract Archival

9. All sample extracts are to be archived in 4.0-mL vials in the sample extract refrigerator for a minimum of 40 days.
10. For each sample, a portion of the extract to be analyzed is to be transferred to a 2.0-mL vial lined with a 500uL micro-insert if necessary.
11. All vials are to be labeled with the following:
    a) Method ID (8082) & Matrix
    b) Sample Reference Number
    c) Extraction Date
    d) Extractionist Initials

J. Sulfuric Acid Cleanup for PCB Analysis (SW-846 Method 3665)

1. Sulfuric acid cleanup is performed to remove many of the contaminants that may interfere with an effective analysis of the sample.
2. If any sample of an analytical batch must undergo acid cleanup, then a portion of the associated QA samples must also undergo cleanup and analysis in the same manner as the samples.
3. Take a portion of the concentrated extract, approximately 1.0ml, and transfer to an empty 4.0ml vial.
4. Add enough concentrated sulfuric acid to the 4.0ml vial to make a 1:1 solution.
5. Cap the vial and shake vigorously for approximately 30 seconds.
6. Allow the solution to separate. The hexane layer will be on top, while the sulfuric acid layer will be on the bottom.
    a. It may be necessary to centrifuge the 4.0ml vial to achieve separation.
    b. If the extract layer is still dark after separation occurs, it may be necessary to repeat the cleanup step.
7. Once the layers have separated, transfer the top layer to an autosampler vial containing a micro insert. Label vials as described in Section IX.I.

K. Sulfur Cleanup with Copper Powder (SW-846 Method 3660B)

1. Transfer ~1.0-mL sulfuric acid cleaned extract to a 2mL vial and add copper powder until it reaches ~0.5mL in the vial.

a) If copper powder does not have a bright shiny appearance it should be treated with dilute nitric acid, rinsed with DI to remove all traces of acid, rinsed with acetone and dried under a stream of nitrogen. This treatment is to ensure that the copper powder is very reactive.

2. Cap the vial and shake vigorously for 1 minute.
3. Allow the phases to separate for at least 10 minutes to ensure that all of the fine copper powder settles out of the extract.
4. Transfer a portion of the extract to a 2mL vial with a 500uL micro-insert and retain the 2mL vial with the copper powder and extract with the archived extracts.

L. Analysis

1. A single-column analysis is performed using a 0.32mm ID Restek Rtx-CLPesticides capillary column (or equivalent).
2. HP G1701BA software is employed to operate the GC/ECD and acquire/store raw data on the 608_ECD computer.
3. GC operating conditions are determined by the analyst and recorded in the following places:

e) Gas flows are recorded in the instrument maintenance log.

f) Temperature, pressure and injection programs are all saved as acquisition method AQ-8080. These conditions are printed and filed with the calibration method and chromatograms after a new calibration method is created.

4. Established operating conditions are used for both calibration and sample analyses.
5. A hexane blank should be injected prior to beginning the initial calibration to assure that the column is free from any interferences which may cause carry problems with integration and to assure that there is not carryover from previous analyses.
6. All analyses of calibration standards, LRBs, LFBs, LFMs, LDs, and field samples must be recorded in the “ECD Run Log” file located on the 608_ECD computer.

M. Dilutions

1. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes.
2. Dilutions are made using hexane and must be re-analyzed with all necessary calibration verification requirements being met.

N. Confirmation

1. If analytes are detected in samples at levels near or above the reporting limit, the sample must be quantitatively analyzed on a second, dissimilar column and meet the same RT and calibration criteria as the initial analysis.
2. If the confirmation result is significantly higher (e.g. >40%) and no anomalies

are noted, report the higher result and qualify the disparity between results.
3. Confirmation may not be necessary if the composition of the sample matrix is well established by prior analysis.

O. Percent Solids

1. Dry weight, used for soil calculations, is determined by measuring the percent solids of the sample.
2. The BOD/Solids analyst determines percent solids using EPA Method 160.3.

X. Calibration

A. Standards

1. All calibration standards are recorded in the ECD Standards log at the ECD workstation. Standards opened and used in the extraction lab are recorded in the Extraction Lab Standards log.
2. Method 8082 is used to quantitate a total of 8 different PCB aroclors, as listed in Section I.
3. A standard containing a mixture of Aroclors 1016 and 1260 will include many of the peaks represented in the other Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of these two Aroclors should be sufficient to demonstrate the linearity of the detector response with the necessity of performing initial calibrations for each of the Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent one of the Aroclors.
4. A minimum of five calibration standards, each containing an equal amount of Aroclor 1016 and Aroclor 1260, should be prepared. These calibration standards should also contain the surrogate standard at different levels, as to calibrate the detector for surrogate analysis. If a quadratic model is used to calibration rather than a linear model, a minimum of six calibration standards must be employed. A 20ppm Primary Dilution Standard (PDS) containing Aroclors 1016 and 1260 is prepared by dilution of the purchased PCB and surrogate mixes with Hexane. The PDS is then used to prepare calibration standards by further dilution with Hexane.
   a) Typical 1016/1260 calibration standards are 0.2ug, 0.5ug, 1.0ug, 2.0ug, 3.0ug, 4.0ug and 5.0ug.
5. Single standards of each of the other six Aroclors (1221, 1232, 1242, 1248, 1254, 1268) are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to demonstrate the linearity of the detector, the single standards of the remaining Aroclors can be used to establish a single point calibration factor for each Aroclor. A 2.5ug standard of each of the remaining Aroclors should be prepared and be analyzed prior to the analysis of any QA or field samples. Typically, they are analyzed immediately after the five-point 1016/1260 calibration standards have been analyzed. These standards are used to screen for Aroclors based on pattern and peak retention time. If any Aroclor is recognized in a sample, the following procedure is to be

followed for quantitation.

a) Use the 2.5ug standard to estimate the concentration of the target analyte in the sample.

b) Adjust the sample so that the concentration in the extract is ~2.5ug.

c) Inject the 2.5ug calibration standard along with a standard at the quantitation limit (0.5ug) and create a two-point calibration curve using 4-6 of the largest peaks.

d) Inject the adjusted sample and calculate its concentration.

6. If deemed necessary for a specific project, a full calibration 5+ standard calibration may be employed.

B. Initial Calibration

1. An external standard calibration procedure is used for initial calibration.
2. Linear Calibration Using the Response Factor
   a) The HP 1701BA Environmental Data Analysis software is used to determine the calibration factor (CF) and relative standard deviation (RSD) for each analyte from a minimum of five calibration standards.
   b) If the RSD is less than or equal to 20% over the calibration range the response factor may be used to determine sample concentrations.
   c) If the RSD exceeds 20% or at the discretion of the analyst, another calibration method listed below may be used.
3. Linear Calibration Using a Least Squares Regression
   a) The HP 1701BA Environmental Data Analysis software is used to perform a linear regression of the instrument response versus the concentration of a minimum of five calibration standards.
   b) A weighted least squares regression may be used if replicate multi-point calibrations have been performed. For all other instances an unweighted least squares method should be used.
   c) The analyst should not force the line through the origin nor include the origin as an additional calibration point. The intercept is to be calculated from the five data points.
   d) The regression calculation will generate a correlation coefficient (r) that is a measure of good fit. When r is greater or equal to 0.990 this calibration method may be used to determine sample concentration.
4. Non-linear Calibration Using a Quadratic Model
   a) The HP 1701BA Environmental Data Analysis software is used to perform a quadratic calibration of the instrument response versus the concentration of a minimum of six calibration standards.
   b) The curve may not be forced through the origin nor may the origin be included as a calibration point.
   c) Weighting using a quadratic calibration model may significantly improve its accuracy.
   d) A coefficient of the determination greater than or equal to 0.990 indicates that the curve may be used to determine sample concentration.
   e) The quadratic model is not to be used to compensate for detector

saturation at higher concentrations or to avoid proper instrument maintenance.

5. Whichever calibration option is employed, analyte and surrogate concentrations must fall within the calibration range in order for calculations to be valid. Dilutions may be necessary in order to bring analyte concentrations within this range.

6. Individual analytes and surrogates within the method may be calibrated using any of the three methods described above.

7. If initial calibration does not meet acceptance criteria the following steps need to be taken:

a) Check to be sure that there are no errors in the calculations or preparation of calibration standard solutions. If a standard is found to be prepared wrong a new standard should be made and injected.

b) Examine chromatograms for interfering peaks and integrated peak areas. If coelution with contaminant peaks is an issue, instrument conditions may need to be changed to achieve better separation or new calibration standards may need to be made with new reagents.

c) Check instrument performance. If an instrument performance problem is found, correct the problem and re-analyze the initial calibration standards.

d) If no instrument problem is found and reanalysis of initial calibration standards is unsuccessful, new calibration standards must be made.

C. Calibration Verification

1. Before analysis of samples and extracted QA can begin the initial calibration is to be verified by the analysis of a calibration standard. For Aroclor analyses, the calibration verification (CV) standard should be a mixture of Aroclor 1016 and Aroclor 1260. If the calculated concentrations of the analytes in the CV are within +/- 15% of the initial calibration, then the initial calibration is considered still valid.

2. CV standards must also be run at the beginning of each 12-hour analytical shift or after each group of twenty samples (including blanks, LFBs and other QA samples), whichever is more frequent. It is recommended that a CV be run more frequently to minimize the number of repeat injections.

3. CV analyte concentrations must fall within +/- 15% of the initial calibration for the calculated value of preceding samples to be considered accurate. If the CV falls outside of the acceptance limits, all samples back to the last passing CV must be reanalyzed.

4. A closing CV must also be analyzed at the end of each analytical shift.

5. If initial calibration standards are analyzed at the beginning of a sequence with a valid calibration resulting, the calibration does not need to be verified until the 12-hour analytical shift beginning 12-hours after the first calibration standard was injected or 20 samples have been injected, whichever is less.

6. If calibration verification results fall outside of acceptance limits, a new standard curve must be prepared unless instrument maintenance corrects the

problem.

XI. Data Reduction

A. Aqueous calculations

1. Calculated analyte masses in micrograms (ug) are divided by the volume of sample in liters (L) resulting in calculated sample concentrations in ug/L.
2. Concentrations may also need to be adjusted for dilutions by multiplying the final concentration by the dilution factor.

B. Soil calculations

1. Soils are reported out as a dry weight concentration.
2. Cs = Cex/(Ms*(PS/100) where;
    a) Cs = Concentration in the sample (ug/Kg, dry)
    b) Cex = Calculated mass in ug of analyte in the final extract
    c) Ms = Mass of the sample in Kg
    d) PS = Percent solid
3. Concentrations may also need to be adjusted for dilutions by multiplying the final concentration by the dilution factor.

XII. Data Recording

A. Locating the 8082 Chain-of-Custody and extracted samples

1. The Chain-of-Custody for extracted 8082 samples will be found in the bins dedicated to methods of the Organic Department labeled “8082”.
2. Extracted samples are located in the Organics Department refrigerator found in the extraction lab.
3. Enter the individual sample information found on the Extraction Log sheet into the Excel Run-log (“ECD”).

B. Target Compounds

1. If analyte concentration is below the reporting limit, report “< (Reporting Limit)”
2. As long as it is within the calibration curve, report that value. Otherwise, dilute the sample and rerun OR add additional standards to the calibration tables. If a target compound is present, then a confirmation process must be done.

XIII. Data Archiving

A. Hard Copies

1. All QC packets from method analyses are to be kept for 5 years.
2. QC packets are separated by method and kept in the filing cabinet near the

608 workstation or in banker boxes (the most recent QC packets are kept in the filing cabinet, space permitting, while all others are kept in folders in labeled banker boxes beneath the workstation).

3. Only the previous and present year QC data are kept on the premises. All other QC data is kept in the Endyne storage facilities.

4. Hard copies of all chromatograms are saved as data packets by order ID with all other Organic data packets.

B. Electronic Backup

1. An electronic backup of all new data files should occur at least once a month.
2. Data is archived on the Unit C computer in the ECD backup folder.
3. Annually, data is backed up on CD-R.

XIV. Instrument Maintenance:

A. Recalibration

1. In addition to recalibration being necessary when calibration verification does not meet acceptance criteria, significant maintenance activities or hardware changes may also require recalibration.
2. Maintenance that should not automatically require recalibration:
   a) Changing septa; compressed gas cylinders; syringes; moisture, hydrocarbon, or oxygen traps; split seals; column fittings; inlet liners.
   b) Other procedures include breaking off or changing a guard column or cleaning the inlet.
   c) Whenever such procedures are performed, the analyst must demonstrate that the results for a calibration verification standard meet the acceptance criteria.
3. Maintenance that automatically requires recalibration:
   a) Changing, replacing, or reversing the column; and replacing the injection port.
   b) Changing or replacing the column will also require that the retention time windows be redetermined.

B. Maintenance Log

1. In the Instrument Log, enter any problems that have arisen with any methods and how the problem was solved. Also log in any changes made to the instrument (i.e. gas flow changes, column changes, injection port changes, etc.).
2. Each entry must be accompanied by the Date and Analyst's Initials

C. Standards Log

1. Whenever any standard is opened or made up, enter the date, analyst's initials, source of standard, type and concentration, and detailed steps as to how the standards were prepared.

D. Maintenance

1. General
   a) The Merlin Micro Seal is good for more than 2000 injections. However, if the traditional septa are used, they must be changed every 30 or so runs.
      (1) Occasional inspection of the microseal for coring or cracking is recommended.
   b) Replace injection port liner every 100-150 injections; whenever standard degradation is evident
   c) Replace injection port orange O-Rings if dried and cracking
   d) Replace column ferrules when cutting the column
2. Injection Port Liner Cleaning
   a) Rinse with $MeCl_2$
   b) Rinse with HNO_3
   c) Rinse with HCl
   d) Rinse with DI
   e) Rinse with MeOH
   f) Dry with N_2
   g) Rinse for 30 seconds with silanating agent
   h) Rinse with Toluene
   i) Rinse with MeOH
   j) Dry with Nitrogen
3. Changing a Column
   a) Lower all GC temperatures (oven, injector, and detector) to < 70°C
   b) Remove the detector end of the column first
   c) Immediately cap the ECD with a plugged ferrule nut
   d) Plug the detector end of the column with and injection port septa
   e) Remove the injector end of the column and plug it into the septa from Step “d”
   f) Remove the old column from the oven and install the new column by reversing Steps “a” – “e”

XV. Trouble Shooting:

A. No Signal

1. Make sure that the column is intact and has the proper flows
2. Make sure that the proper detector is selected in the method that is loaded

B. Noisy / High Baseline

1. Using an air duster, check for leaks into the system by spraying at system joints from injector to detector. If a leak is present, there will be sharp spikes in the signal.
2. Double check column insertion distances. The column on the detector end should extend about 6.5 cm from the ferrule, and the column on the injector end

about 2mm.

a) The best way to achieve these distances is by installing the column with the new ferrules, but leaving them loose enough to push/pull on the column. Then, push the column all the way in and then back it off about 2mm. Then tighten the ferrule.

3. Cap detector and bake for 3-6 hours. It may be necessary to raise the temperature to 350 or 400°C. DO NOT LEAVE AT 400°C OVERNIGHT. After this, the signal should fall and remain stable.

4. If after baking the signal is still too high and there are no leaks, then it is probably time to remove the detector and ship it out to be professionally cleaned thermally and chemically.

5. If after baking, the signal seems acceptable, then re-install the column. If the signal goes up and remains high, then there may be contamination on the column. This contamination should be removable by baking the column (removed from the detector) at 310 - 320°C for 3-4 hours.

C. High or Low Column Flows / Head Pressure Not Adjustable

1. Split vent lines may be contaminated with sample waste. To resolve this problem, rinse the purge lines with MeOH and blow dry.

2. The column may be plugged. Check the detector and injector ends of the column (using XIII:3C) for blockage

D. No Head Pressure

1. Make sure the injector column fittings are tight.
2. Make sure the “Total Flow” valve is open.

E. Reduction in Response / Coelution of Certain Analytes

1. There may be active sites in the injector / column. Try changing the injection port liner, gold plated seal, septa, or removing 1-2 loops of column.

2. Check column flow. If flow is too high or too low, coelution and/or tailing may occur.

XVI. Method Modifications:

1. Sec. IX.D. Method 3550B calls for the solvent to be decanted through filter paper after sonication. Endyne filters through muffled sodium sulfate and glass wool to remove particulate and further dry the extract.

XVII. Bibliography/References:

Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0. EPA SW-846 NTIS, Dec. 1996.

Method 8000b Determinative Chromatic Separations, EPA SW-846 NTIS, CD-

ROM Version 2, Dec. 1997

GC Trouble Shooting Guide, 3rd edition, Phenomonex, August 1999

HP 5890A Series II Reference Manual, fifth edition, Hewlett-Packard, Sept. 1991 Publication # 05890-90270

HP 7673 Automatic Sampler, forth edition, Hewlett-Packard, June 1992 Publication # 07673-90185

Operating Hints for Split/Splitless Injectors, Restek, 1992. Bellefonte, PA.

A Guide When Injecting Dirty Samples, Restek, 1994. Bellefonte, PA.

XVIII. Revision History:

A. from revision #7 revision #8

1. Added Bibliography/Reference section; Added Revision History section; Added sign off sheet.
2. Edited typos; formatted tab sets and spacing; updated table of contents
3. Added laboratory safety requirements.

B. Revision #9

1. Defined MDL determination as analysis of only seven samples with Standard Deviation multiplied by 3.14
2. Added PCB in Oil sample preparation
3. Defined the point in Internal Standard addition as after the sample extract has been concentrated and transferred to analytical vial.

C. Revision #11 (Mark Plasse)

1. Complete revision of extractions and cleanups to include all methods used in the lab. Also added sonicator calibration instructions.
2. Clarified calibration section to include all linear and non-linear calibration techniques, calibration verification.
3. Revised summary and introduction as well as reporting limits and QA sections.
4. Expanded Quality Control section to include more detailed information about the acceptability of QA/QC samples and surrogates as well as protocol for out of control QA/QC.
5. Defined calibration line and QC samples to contain a mixture of Aroclors 1016/1260 rather than Aroclor 1254.
6. Defined single point Aroclors to be analyzed after calibration and prior to analysis of field samples.

7. Eliminated use of internal standard.

I acknowledge that I have read this Standard Operating Procedure and agree to adhere to the Safety and Quality Control policies outlined within.

Name	Date

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ICP
Standard Operating Procedure

EPA Method 6010B

REVISED: April 4, 2005

Revision #: 3

Technical Director: _____Date : _____
Harry B. Locker Ph.D.

QA Officer: _____Date : _____
Mark L. Fausel

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I. Overview:

- Inductively coupled plasma-atomic emission spectrometry (ICP-AES) may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, and domestic and industrial wastewater. In addition, TCLP extracts, soils, sludges, sediments and other solid wastes may be analyzed. This SOP is based upon EPA Method 6010B. The analysis described in this method involves multielemental determinations by ICP-AES using either a sequential or simultaneous instrument. The instruments measure characteristic atomic-line emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the line spectra are monitored at specific wavelengths by a photosensitive device. Photocurrents from the photosensitive device are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of the analytes.

II. Safety:

- Gloves, goggles, and lab coat should be worn when handling all samples, reagents, and standards. Acids and uncovered digesting samples should be handled in the fume hoods. An exhaust fan is installed above the ICP to remove emissions. The toxicity or carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucous membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The inductively coupled plasma should only be viewed with proper eye protection from the ultraviolet emissions. It is the responsibility of the user of this method to comply with relevant disposal and waste regulations

III. Waste Disposal:

- Liquid waste from the drain lines of the ICP is collected in a 10 gallon plastic container with a cap. When it’s full, it is disposed of down the sink with copious amounts of water.

IV. Sample Collection, Labeling, Preservation, and Storage:

A. Collection:
Samples are collected in plastic bottles of various sizes.

B. Labeling:
All samples are assigned a number, plus a unique identifier suffix to indicate preservative and bottle type.

C. Preservation:
Upon arrival at the lab, water samples are preserved with a few drops of concentrated Nitric Acid to a pH of <2. Sample bottles preserved with nitric acid are labeled with a red suffix.

D. Storage:
Water samples are stored at room temperature in drawers by the RO system on the first floor. Soils and sludges are stored in the fridge on the first floor. Digested samples are stored in labeled drawers beneath the metals lab bench.

V. <u>Equipment and Supplies:</u>

- Volumetric flasks for making standards and diluting samples.
- Pipettes for digestions
- Leeman Labs Inc. ICP PS 1000
- Water recirculator, exhaust fan, Argon gas
- Leeman Labs DRE computer software
- A variable-speed peristaltic pump is required to deliver both standard and sample solutions to the nebulizer.
- Analytical balance, with capability to measure to 0.1 mg, for use in weighing solids, for preparing standards, and for determining solids in digests or extracts.
- Block digester capable of maintaining 95^0C
- 50 ml plastic containers for the block digestor, plastic watchglasses and caps
- Timer for turning block digester on and off
- Cellulose air filters
- Smear Tabs

VI. Reagents and Standards:

- Concentrated Nitric acid
- Concentrated Hydrochloric Acid
- 30% Hydrogen Peroxide
- Multielement and single element standards
- Concentrated Perchloric Acid

NOTE: All incoming standards, reagents, dry chemicals, and acids shall be marked with date of receipt, date of expiration, and date of opening. Dry chemicals and concentrated acids shall be used within 5 years of the date of receipt.

VII. Interferences:

A. Spectral Interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
B. High concentrations of Iron may interfere with other elements.
C. Aluminum used in sludges can interfere when analyzing Arsenic by ICP
D. An Interelement Correction Std. is run annually.
E. To perform an Inter-element Correction:
   1. Under “calibration”, “inter-element correction” if “analytes” on the left are not asterix-marked, select these and continue hitting enter until all “interferents” are 0.00000.
   2. Under “calibration”, “standards”, to the far right is an “IEC” column, enter the concentration of each interferent. Enter zero for those elements not being looked for as interferents.
   3. Under “protocol”, “set values”, be sure the interelement correction column says “Y”
   4. Under “protocol”, “on/off elements”, select all elements in the protocol.
   5. Run only single elements and each should be between 200-500ppm.
   6. Place the standard in large cup in slot two and send the auto-sampler there.
   7. Hit F6, select “IEC” enter the line (the interference you are running) select “2” replicates.
   8. Go to “inter-element correction” page to see if there are any hits.
   9. When running samples, if you want the inter-element correction to be done by the software be sure the inter-element correction column says “Y” (see step 3).
F. The ICP is very temperature sensitive. A temperature change of a few degrees an hour will interfere with results, especially for Ba and Al.
G. An interference check standard is analyzed at the beginning of each analytical run. This is done to verify the Interelement and background correction factors. Results should be +/-20% of the true value.

VIII. <u>Quality Control:</u>

A. (See Table A in the SOP SOP for more detailed definitions of the following)
   1. Demonstration of Capability (DOC). Each new analyst performs a set of four mid-range LFBs. The LFBs should be prepared as one sample, then split into four aliquots. The percent standard deviation and average percent recovery are determined for each analyte. Each must be less than 20% to ensure proper capability of the analyst for that method. This process is repeated annually for each metals analyst.
   2. Method Detection Limit (MDL).
      a. An MDL is run annually for each element, at 2-5 times the lowest reported value. The final calculated MDL value must be at least ½ the reported value **and** the MCL. MDL’s should be digested and also non-digested.

3. Calibration Check/Verification (or Instrument Performance Check – IPC). The concentrations of the calibration verification shall be varied within the established calibration range.
4. Laboratory Reagent Blank (LRB).
5. Laboratory Fortified Blank (LFB or Independent QC)
6. Laboratory Fortified Matrix Spike (LFM).
7. For all determinations the laboratory must analyze a calibration blank and line check (IPC) at the start of the sample run, after every tenth sample (or more frequently, if required) and at the end of the sample run. The IPC's must be +/- 10%.
8. The calibration blanks can not be greater than 1/10 of the MCL (regulatory limit), or greater than 1/10 of the lowest measured sample in the batch.
9. Percent Recovery for LFB's must be +/-15%.
10. Interference Check. Prepared to contain known concentrations of interfering elements that will provide an adequate test of instrument correction factors. Analyze at the beginning of each analytical run. Results should be +/- of the true value.
11. Duplicate samples are run every batch or every 20 samples
12. LFM samples are run every 20 samples or when results are questionable. Percent recovery for LFM's must be +/-30%.
13. LFM's are acceptable as Independent QC's.
14. If data does not meet the criteria outlined above or in the sample preservation section, it may be used, but MUST be noted on the final client report.

IX. Analytical Procedure:

A. Sample Preparation

1. Drinking water sample preparation is dependent on the turbidity. If the turbidity is < 1.0 the samples are analyzed "as is". If the turbidity is > 1.0 the samples are analyzed "total".
2. For sample preparation and digestion, see Appendix 1

B. Data Acquisition

1. Data acquisition is done using Leeman Lab DRE software
2. Overview:
   a. The Leeman Labs Software is "Command Central" of ICP operations. So in order to operate the ICP, the Leeman Software must be currently running. You can tell that you are in the Leeman Software when you see on the monitor a black background with a large blue box at the top of the screen and a small blue box at the bottom; depending on how the computer was left, the Main Menu should be present (type F1 to get to Main Menu).
   b. Start the software if it is not already running by typing **PS** at the C:\ICP> prompt. Make sure you are in the \ICP subdirectory before starting the software. The computer is configured to store the raw data on a compressed drive (H:\).

The data folders are in the H:\FOLDERS subdirectory. A backup of the current protocols and macros can also be found on the H:\ drive.

3. The Lay of the Land
   a. There are 10 keys on the keyboard that have dedicated functions. They are also known as "Hot Keys". No matter what menu you are in, these functions can be accessed. These keys are found at the top of the keyboard and are described below:

      F1: Menu - returns any current screen to the Main Menu
      F2: Macro - allows the execution of a "Macro", which is a set of automated commands
      F3: Print Screen - prints the currently displayed screen
      F4: Report - allows custom printing or saving of data files
      F5: Update - used to update lines for analysis
      F6: Standard - used to read calibration standards
      F7: Check Standard - used to read a check standard
      F8: Sample - used to run a set of samples
      F9: Scan - used to scan a sample
      F10: Stop - stops any current function
   b. In addition to these dedicated keys, many of the alphabetic keys serve as function keys depending on the menu or screen. In every menu or screen option, all letters will be the same color except for one. Push the key corresponding to the letter in the alternate color to choose that particular option.

C. Igniting the Plasma
   1. The plasma should run for 40 minutes prior to use in order to reach thermal equilibrium. There are 6 main interlocks as indicated at the upper right corner of the plasma side of the ICP. These interlocks include Power, Plasma Door, Argon, Water, ICP Covers, and Spray Chamber Guard. If any of these interlocks is lit, the plasma will not ignite. Argon supply must be between 85 and 90 psi. If the Argon pressure falls below 75 psi, the Argon interlocks will begin to flash. If the pressure falls below 60 psi, the plasma will self-extinguish. The Argon pressure should never go above 120 psi.
      a. Automatic Ignition
         i. Turn on plasma side of ICP by pressing the green button.
         ii. Make sure all guards and covers are in place.
         iii. Turn on Argon and check pressure (90 psi).
         iv. Turn on fume hood by flipping the single light switch behind the liquid argon tank.
         v. Turn on Water recirculator.
         vi. Lower Autosampler tip into DI.
         vii. Check pump tubing integrity.
         viii. Raise tension bar on the pump.
         ix. Choose **I**CP menu.
         x. Choose **O**peration
         xi. Choose **C**oolant, Turn it On.

xii. Press Auto**S**tart to start the plasma.

xiii. After AutoStart completion, the message bar will indicate "Autostart Completed

b. Manual Ignition - **only to be used by experienced operators**!

i. Perform steps 1-10 from above

ii. Choose Nebulizer and Select On

iii. Wait 30 seconds

iv. Choose Nebulizer and Select Off

v. Choose Coolant and Select On

vi. Choose Power and Select On

vii. Press I to Ignite the plasma

viii. Choose Nebulizer and Select On

ix. Choose Pump Rate and Select On

x. The plasma should now be ignited

D. Open Protocol & Folder

1. A Protocol is a Leeman data file that contains information concerning analytical lines, integration parameters, calibration and check standards, and other related data. A Protocol must be created and opened before any analysis can begin. Once a Protocol has been created, it can be saved and opened at a later date.
2. In order to create a Protocol
   a. From Protocol, select Get and enter a Protocol name. Any name can be used, but for month to month general analysis, a Protocol indicating the month the Protocol is used may be a good idea. For example, Protocol name 02032007 would refer to a Protocol that underwent a new calibration in Feb. 2003. If you choose a Protocol name that does not exist, you will be asked if you want to create that Protocol; if you do, enter Y for Yes.
   b. Return to the Main Menu
   c. If you are creating a completely new Protocol, choose Element Selection. If not, proceed to e).
   d. Enter the atomic symbol for the first element of the list for which you will want to analyze. For example, enter Ba for Barium.
      i. After you enter the symbol, a list of available lines for the selected element will appear. Each available line will be indicated by the atomic symbol combined with a number. One or two lines will have an asterisk to the left. Most, if not all, lines will have a question mark to the right of the symbol.
      ii. The second major column contains the wavelength, in nm, of the line. The third column contains either a “I” or “II”; “I” indicates that emission for that lines originates from an excited atom; “II” from an excited ion. The fourth column contains the relative intensities of each line.
3. Generally, you will want to choose the preferred line as indicated by the asterisk. If more than one line has an asterisk, choose the line with an asterisk and the highest relative intensity. A line is selected by entering the number that corresponds to that line. So from the list for Ba (Ba1, Ba2, Ba3), enter 1 and the line Ba1 will appear on the left half of the screen. For Cr, the lines Cr3 and Cr4 are both preferred lines

and have the same relative intensities, therefore, you need to select a line that is not near any other line or depend on past experience. The line Cr4 tends to be the preferred line in an extended list of lines for Chromium.

4. If your chosen line has a question mark next to it, you must perform a Peak Wavelength on that line before using it. Peak Wavelength tells the ICP where to find that line in the Optics Box.
5. Continue entering lines until you have entered all the desired elements.
6. Return to the Main Menu.
7. Select Set Values.

E. Set Values contains integration parameters, sample uptake time and sample weight and dilution factors.
   1. For most lines, choose 3 Integrations. Use 2 Integrations for Mo and Sb.
   2. Uptake time for all lines, choose a time of 40 seconds. The ICP will draw up sample for 40 seconds before it begins analyzing it.
   3. Scan integration time should be set to 1 tenth of a second for all lines.
   4. If you will be using Weight and Dilution factors, enter Y for yes for each option. More information on these options will appear in the Sample Analysis Section.
   5. The Peaking Line is a line that is scanned to find the optimum viewing position in the plasma. The line Mn1 is a good line to use because it is centrally located within all other lines. Therefore, the line Mn1 should be included in Element Selection regardless of whether you will be testing for Mn. The Peak Both Routine will be discussed in Section D1.
   6. For all lines, but Mo and Sb, Background Integration = Peak should be N for No.
   7. Return to the Main Menu.

F. Choose On/Offs, Times, Gains.
   1. Time for all lines except for Mo, Sb and K should be 3; for Mo, Sb and K use 10.
   2. The On column indicates whether a line is on or not. You will only be able to analyze for a particular line if it is on. All lines can be turned on by selecting on and entering Y for yes. If you only want to turn on some lines, push the insert key and use the cursor direction keys to move up and down the list. When the on column next to the desired element is highlighted, enter Y for yes and continue down the list.
   3. Return to the Main Menu

H. Internal Standards compensates for a group of elements that shift together. However, this option is not used currently.

I. Choose Limits. Limits can be used to flag a sample with a "L" if the result is below the detection limit, or with an "H" if the result is above the calibration line.
   1. Push the Insert key and use the cursor keys to move around the columns.
   2. Enter detection limits in the Min column keeping within the same units that you will use on the calibration standards page.

3. Enter values corresponding to 110% of the highest calibration standards for the Max values.
4. Return to the Main Menu.

J. Maintenance deals with file and disk management.

K. Choose Auto-sampler Menu
1. Choose Setup
2. Select Update cups
4. Enter "1" for "UI". "UI" is the Update Intercept Cup. The Intercept is the calibration acid blank and should be found in Standards Rack Cup Position 1.
5. Update Slope standard should be the highest standard of the calibration curve. The normal monthly calibration usually consists of the blank plus 3 standards, so the high standard can be found in Cup 4. Therefore, enter 4.
6. Frequencies allow you to have the ICP automatically perform Update and Peak routines. Leave UI and US at 0. Enter 5 for Peak.
7. Choose Check. Enter 10 for C1 and C2 and then push the Escape key when done. These values have the ICP automatically read the Acid Blank and Check Standard every 10 samples.
8. Leave Halt as No. Placing Halt as Yes would terminate all analyses even if only 1 line of a list should fail.
9. Set the rinse time to 40 seconds. The Autosampler tip will return to the rinse tank and wait 40 seconds between every sample and standard.
10. Set Cups per rack to 60 since there are 60 cups per sample rack.
11. Return to the Main Menu.

L. Choose ICP Menu
1. Choose Operation
2. A summary of plasma conditions for aqueous and organic samples appears in the Appendix. Enter the appropriate values. Keep in mind that the monthly Calibration is for aqueous samples.
3. Choose Peak Cup and enter 6. Based on this value, a 10 ppm Mn solution will have to be in Standards Rack Cup 6 for the Peaking Routine (finding the optimum plasma viewing zone).
4. Autostart plasma/Ignite plasma/Extinguish deal with plasma operation. The first 2 options were detailed in section IB. Extinguish, obviously, extinguishes the plasma.
5. Do not Void Interlocks. The Interlocks prevent plasma ignition if any hazard is present.
6. Unfreeze Write Parameters do not relate to the Protocol. This option allows you to freeze or unfreeze the operating parameters as currently set. Write parameters copies the parameters into the Protocol.
7. Tip to rinse sends the Autosampler from any current position back to the rinse tank.
8. Return to the Main Menu.
9. Peak Both corresponds to peak X, Y, Both on Operations page and is part of the peaking routine, described in Section D1.

L. N. Choose Calibration Menu.
   1. Select Standards
   2. The resulting table allows you to enter units (ppm, ppb, mg/kg, etc.) and standard values.
   3. Pushing the Insert key and using the cursor keys, you can move to any cell and enter the appropriate response.
   4. UI should be 0 for all lines since UI because you will be using an acid blank as the Update Intercept.
   5. US should be equivalent to the highest standard.
   6. S1 is, again, the acid blank and should be 0 for all lines.
   7. Enter the desired standard concentrations for all lines for the remaining standards. Press the page down key if not all lines appear on the screen.
   8. Return to the Main Menu when complete.
   9. Choose Check Standards.
   10. Press the Insert key and use the cursor keys to move between cells.
   11. In the C1 column, enter the detection limit for each line. If, when reading check standard 1 (Acid Blank), the resulting concentration for a line is outside the range of $\pm$ the MDL, the result will be flagged.
   12. In the C2 column, enter concentration equivalent to the midpoint of the calibration curves and enter 5 for each check standards $\pm$% column. If, when reading the check standard, the result is not within 5% of the correct value, the result will be flagged.
   13. Return to the Main Menu when done.
   14. Spike quantities allow you to enter spike concentrations in a similar manner as calibration standards. However, the cup macro that is supposed to control spike % recovery does not appear to work currently and, so, is not used.
   15. Line Calibration allows you to look at calibration curves and accept them. This area will be detailed later in the Calibration Section II.
   16. Accepted Values lists the currently accepted calibration constants.
   17. Reset allows you to reset calibration values.

M. Choose Data Output
   1. Open Folder allows you to open a folder. A Folder stores data about scans and sample analysis and is not part of creating a Protocol.
   2. Folder names containing Calibration data should be the same as the Protocol name. Folder names for samples should be the date on which the samples were analyzed. Weekly QC Folders can take the name of the current date.
   3. Folder Maintenance allows data and file disk control and will be in Disk Management.
   4. Choose Specify Report. Specify report dictates what and how sample data is reported.
   5. Choose Edit.
   6. Using the cursor keys, turn everything in the Real Time column to Y except wavelength and Intensities. A Real Time Report is a report that is generated as data is acquired.
   7. Return to Main Menu.

N. Creating a Protocol - Summary.

All necessary information for creating a protocol should now be entered. Any changes to a Protocol are automatically saved to the hard disk upon making the change, so be careful not to change something in a Protocol that you do not want to change. Many operating conditions stated above are automatically performed by the Macro "Setup". However, "Setup" is designed to work with the monthly curve with the name in the format MMDDYY (such as 020303 – Feb. 3, 2003). Some deviations may occur if this macro is applied to an alternate Protocol.

O. Peak Both

1. The Peak Both routine helps to find the best viewing position within the plasma using a high concentration of a particular line. A Peak Both routine, should occur at the beginning of each day before any sample analysis occurs.
2. For aqueous samples, the Peak Both Solution is 10 ppm Mn. For organic samples, the Peak Both Solution is 20 ppm Fe.
3. The procedure for the routine follows.
   a. Select ICP Operations page.
   b. Verify that the Peak Cup is 6. If not, choose Peak Cup and enter 6.
   c. Place a Standards vial containing the Peaking Solution in Standards Rack Cup 6.
   d. Push B under Peak X, Y, Both.
   e. The ICP will first perform a Peak Optics routine and then will move to the Peaking Solution.

   f. Observe the resulting X, Y Scans.
      i. The X scans should be centered and symmetrical around the Vertical line within the Scan View box.
      ii. The Y scan should follow the same criteria as the X scans. The Y scans are not normally as neat as the X scans and, so, some judgement calls will have to be made. It is most important that the right side of the Y scan has a consistent downward slope and that the entire scan is centered around the vertical line.
   g. While observing the X, Y scans, also make a mental note of the Intensities underneath each X, Y scan.
      i. Leeman suggests that these X, Y Intensities be within 1% of each other.
      ii. Based on past experience, Intensities within 2-5% of each other tend to be OK.
   h. If the criteria of f) and g) are not met, repeat the Peak Both routine repeatedly until the scans match these criteria as closely as possible.
      i. If either the X or Y tends to look particularly bad, do a peak in just the X or Y direction by pushing X to Peak X or Y to Peak Y.
      ii. Do not perform more than 2 Peak Y routines without doing at least one Peak X or the X direction will be adversely affected.
   i. Push F10 to Stop when the Peak Both routine is satisfactory.

P. Calibrating the Instrument

In order to calibrate the ICP, a Protocol and Folder must be open. The following procedure assumes that a desired Protocol has been created. If there is no Protocol, you will need to create one following the procedure found in Section I C. It is a good idea to recalibrate each month and so the Calibration date becomes an ideal name for the normal monthly Protocol and its associated Folder. The current format is to use the name MMDDYY that corresponds to Month Day Year.

1. If a currently calibrated Protocol does not exist:
   a. Choose Protocol Menu
   b. Get the Protocol that has been created, but not calibrated
   c. Enter a Folder name and create the Folder if it does not exist
   d. Proceed to Step 8 of the next section
2. If a currently calibrated Protocol does exist:
   a. Choose Protocol Menu
   b. Choose Maintenance
   c. Select Copy
   d. Enter the Protocol name that you wish to copy and then enter the name of the Protocol to which you will copy the old Protocol
   e. Get the new Protocol and open a Folder.
   f. Choose the Calibration Menu
   g. Choose Reset
   h. Choose each of the 4 options (Coefficient/New Calibration/Update Standard/Spike) to reset and enter Y for yes to reset each parameter
   i. Return to the Main Menu
   j. Choose Protocol Menu
   k. Choose On/Offs, Times, Gains
   l. Select On and enter Y for yes
   m. Turn off Mo Sb
   n. Return to the Main Menu
   o. Choose Autosampler Menu
   p. Choose Setup
   q. Place a 2% HNO_3 Solution in Standards Rack Cup 1
   r. Select Tip to cup and enter Station 0 Cup 1. The auto-sampler tip should now move to the acid blank.
   s. Press the F9 key.
      i. Choose, but do not enter, an appropriate name such as 2% HNO_3
      ii. Wait 40 seconds
      iii. Press Enter. The ICP will now scan the acid blank for all lines in the Protocol.
   t. Press F10 when the scan is complete.
   u. Return to the Main Menu.
   v. Choose Protocol Menu.
   w. Choose On/Offs, Times, Gains.
   x. Turn off Ba, Ag, Al, K, Na.
   y. Send the auto-sampler to a 1 ppm solution of SPEX Inc QC-19.

z. Wait 40 seconds and press F9 to scan QC-19 for Be, Cd, Cr, Co, Cu, Fe, Mn, Ni, Zn, Ca, Mg, As, Pb, Se.
aa. Turn off QC-19 lines on Protocol On/Offs, Times, Gains page.
bb. Turn on Ba, Ag, Al, K, Na on Protocol On/Offs, Times, Gains page.
cc. Scan a 1 ppm (10 ppm K) solution of Spex Inc. QC-7 by pressing F9 and entering an ID after a 40 second wait.
dd. Scan the highest Calibration standards of sets A, B, C, and D for the appropriate lines. Remember to turn on only the appropriate lines for each solution in the Protocol On/Offs, Times, Gains page. Scan by pressing F9 and entering an ID after a 40 second wait.
ee. When all scans are complete, choose Protocol Menu.
ff. Select Background and Scans.
gg. Cursor down the list and push enter for each available scan. As each scan is turned on, its description will appear to the left of the scan view box. Make sure that all scans are listed. Keep in mind, however, that there will not be peaks for all listed scans because some scans were only for selected lines.

hh. Choose background point(s).
    i. The scans of interest for each line are the 2% HNO_3, the appropriate QC, and the highest Calibration standard of that line. It may be helpful to turn off the other scans by highlighting the scan description and pushing enter.
    ii. Press Z for Zoom repeatedly until you can see the baseline in the greatest detail without the baseline zooming out of the view box.
    iii. For most lines, the baseline will be level on both sides of the peak. For these lines, only 1 background point is necessary. Use the cursor Left/Right keys to move the vertical cursor bar within the view box.
    iv. The best possible background point is when all three scans are at their lowest point together and the scans are relatively flat. Once you have found such a point, press 1 to select that point.
    v. If you cannot find such a point, select a point where all three scans are at their lowest point together and all tend to move in the same direction. Press 1 to mark the point.
    vi. If the background is not level, or if it dips on one side, it may be necessary to choose 2 background points, 1 on each side of the peak.
    vii. Use the Page Down key to advance to the next line.
ii. After all background points are selected, turn off all lines except for those in 1 of the 4 Calibration standard groups.
jj. Make sure appropriate Calibration standards are present.
kk. Execute the Macro "AUTOCAL".
    i. Push the F2 key and enter Autocal.
    ii. For the normal Protocol, enter 5 for the number of standards.
    iii. Enter 3 for the number of replicates.
ll. Instead of calibrating using Macro "Autocal", a calibration can be manually performed using the F6-Standard Key.
    i. Press F6 and the number of the standard to run.

ii. Press Enter to begin with Replicate 1.
iii. Enter the final replicate number, typically 3. The ICP will now read the standard.
iv. Repeat Steps 1-3 for each additional standard.

mm. After the Calibration is complete, press F6 to review the Calibration curves.

nn. Accept the individual curves as appropriate.
i. The curve should be linear and pass through the point (0,0). If the curve extends about 1/2 an inch or more in the negative y direction, a wrong background point was used and it will be necessary to recalibrate with a different background point.
ii. Check the entered concentrations with the calculated Concentrations Logically, these values should be fairly close to each other. Also, the Calculated value for the blank should be less than the detection limit for that line.
iii. The Correlation Coefficient, r, should be at least $\pm$0.995.
iv. The % RSD for each standard should definitely be less than 10 and preferably less than 5.
v. A line can be accepted as Linear, Weighted Linear or Quadratic. A Linear line puts equal emphasis on all points. A Weighted Linear line puts emphasis on the lower end of the line and, so, is better for trace level work. A Quadratic line should only be used when there is a very large dynamic range and must include a blank and 6 standards to be mathematically applicable. For the normal Protocol, all lines will be either Linear or Weighted Linear with a preference for the Linear. Accept lines as appropriate.
vi. After accepting all lines within a solution, analyze QC-7 and QC-19 for the appropriate lines. Each line can only be used if its QC is within 5% of 1 ppm.

oo. Repeat steps hh.-nn. for the remaining solutions.

pp. Repeat steps n.-nn. for Mo Sb, using QC-19, Std. C, and the following protocol values: # integrations = 2, "Background integration = peak:" yes.

qq. If the QC fails for any line, the problem must be corrected and the line re-calibrated until the QC does pass.

Q. Method of Standard Additions

The Method of Standard Additions allows for compensation of chemical interferences by using the sample, instead of 2% HNO_3, as the diluent for the standards. The sample and 2 spikes of the sample are used as the standards of the calibration curve. The first spike should be equivalent to twice the expected sample concentration. The second spike should be equivalent to twice the concentration of the first spike.

1. Create a Protocol with the name of the sample followed by SA. For example, Protocol name 25,009SA would indicate sample number 25,009 which was analyzed by Standard Additions.
   a. Follow the procedure in Section IC for creating a Protocol.

b. On Calibration Standards page, enter 0 for Standard #1, the value of the first spike for Standard #2, and the value of the second spike for Standard #3. These values will change dependent on the spike.
c. There will be no value to enter on the Calibration Check Standards page.
d. Be sure to reset Calibration values on Calibration Reset page.

2. Turn on all lines on Protocol On/Offs, Times, Gains page.
3. Send the Autosampler to the unspiked sample.
4. Press F9 and wait 40 seconds.
5. Enter an ID for the sample.
6. Send the Autosampler to the first spiked sample and repeat Steps 4-5.
7. Send the Autosampler to the second spiked sample and repeat Steps 4-5.
8. Press F10 to stop when all Scans are complete.
9. Go to Protocol Background and Scans page.
10. Select Background points as in Section II B 33.
11. Execute the Macro "Autocal" using 3 Standards and 5 Replicates.
12. Press F6 to review the calibration curves.
13. Review and accept the calibration lines as in Section II B 38; however, remember that Standard 1 is your sample, not a blank, and so may give a calculated value. Also, Correlation Co-efficient, r, of $\pm$0.995 or greater are acceptable for Calibration done by Standard Additions.
14. To retrieve the results of the sample.
    a. Go to Calibration Accepted Values page.
    b. Press F3 to Print Screen.
    c. The negative of the value in the C column is the sample concentration for that line in the units as indicated on the Calibration Standards page.

R. Sample Analysis

In order to analyze samples, a Protocol and Folder must be open. Currently, Folder names are equivalent to the date on which the sample was analyzed.

1. Select Main Menu.
2. Get a Protocol and Open a Folder. Create the Folder, if necessary.
3. Update the lines of interest.
    a. Choose Protocol Menu.
    b. Select On/Offs, Times, and Gains.
    c. Turn on all lines of interest.
    d. Place 0.5% Nitric Acid in Standards Rack Cup 1.
    e. Press F5 and choose Intercept. The auto-sampler will move to the acid blank and read the intercept.
    f. Return to On/Offs, Times, Gains.
    g. Turn on only those lines of interest within 1 standard group.
    h. Place the proper standard in Standards Rack Cup 5.
    i. Press F5 and choose Slope. The auto-sampler will move to the standard and read the Slope.
    j. Repeat f-i for any other standard group necessary.
4. Analyze Check Standards 1 and 2.

a. Make sure 0.5% HNO_3 is in Standards Rack Cup 8 and Calibration Check Standard is in Standards Rack Cup 9.
b. Press the F7 key and choose Check Standard 1. The ICP will now analyze the Acid Blank.
c. Press the F7 key and choose Check Standard 2. The ICP will now analyze the calibration Check Standard.
d. If either Check Standard should fail for any line, you are allowed by the EPA Method to analyze the check standard a second time and use whichever Check Standard passes. If a Check Standard fails both times, samples for that line cannot be analyzed until the Check Standard passes.
e. For failing lines, try performing a complete Update (Intercept and Slope), or just a Slope or Intercept and reanalyze the check standards.
f. If the check standards still fail, Reset the Update Coefficients, perform a complete Update of all lines and reanalyze the Check Standards.

5. Analyze Interference QC.
   a. Make sure Interference QC is in Standards Rack Cup 12.
   b. Press the F7 key and choose Check Standard 5. The ICP will now analyze the Interference QC.
   c. As with previous check standards, the EPA allows this to be analyzed two times to determine its passage.
   d. Once all needed lines pass their check standards, choose Auto-sampler Menu.
6. Choose Rack Entry.
7. Choose Edit and enter a Rack Name. Use any name that you desire.
8. Enter Sample ID, extended ID, weight and dilution factors as necessary.
   a. Pressing the insert key and using the cursor keys will allow you to move from cell to cell.
   b. Cup ID should only include reference numbers.
   c. Extended ID should indicate dilution factors and whether a sample is Dissolved, Total or TCLP and whether it is a Repeat/Duplicate and/or a Spike.
   d. Dilution factors, if used, should be in ml of final Volume.
   e. Weight factors, if used, should be in grams of sample.

   *NOTE, that weight and dilution factors are not currently used.

   f. Cup Macros, entered in the macro column, are not currently used. See the Leeman Reference Manual Revision 2.0 for more information. Check and Help are related Cup Macro functions. To Sequence Cup IDs, choose Range and enter the appropriate Cup numbers and then choose Sequence.
   g. To enter a similar response to a range of extended IDs, select a Range, choose Extended ID and enter the ID.
   h. After all ID information is entered (keep in mind a Repeat/Duplicate and a spike for every 10 samples), press the F8 key.
9. Select Rack 1 and enter the Rack Name.
10. Enter the Cup numbers that you want to read.
11. Repeat Steps 9-10 for Rack 2, if needed.
12. Press F8 again to begin analysis.

a. Since the Macro "Setup" should have set the Check Standard and peak frequencies, these readings should occur automatically through the run.

13. Once analysis is complete, verify that QC Protocol, as detailed later, has been maintained and reanalyze any sample as deemed necessary.
14. If QC Protocol is acceptable and all necessary data has been accumulated, press page eject to print out data.
15. To begin work on a new project, return to Step 1.

S. Shutting the plasma down at the end of the day.

1. Choose Main Menu.
2. Choose Autosampler Menu.
3. Choose Setup.
4. Rinse the sample lines with 25% nitric acid for 5 minutes. This helps to clean the nebulizer.
5. Send the auto-sampler to a DI rinse for 10 minutes.
6. Lift the autosampler out of the DI rinse and allow the pump to run until the lines are dry.
7. Choose Main Menu.
8. Choose ICP Menu.
9. Choose Operation.
10. Push E to automatically extinguish the plasma.
11. Shut off the water cooler.
12. Turn off the argon supply.
13. Shut off exhaust fan and ICP.
14. Release tension bar on the pump.
15. Back up data as detailed in Section XI, Disk Management.

X. Calibration and Standardization:

A. Calibration lines are run monthly for the four groups of metals in the 200.7 Protocol

- Group A: Ba Be Cd Cr Co Cu Fe Mn Ni Zn
- Group B: Ca K Mg Na
- Group C: Ag Al Mo
- Group D: As Pb Se

1. Four points are run for each line (Table B)

Group A calibration points are from a multi-element standard (Stock A) containing the following...

Ba 200.8 ug/ml Be 100.0 ug/ml Cd 100.8 ug/ml Cr 200.0 ug/ml
Co 200.0 ug/ml Cu 200.8 ug/ml Fe 200.7 ug/ml Mn 200.0 ug/ml
Ni 400.8 ug/ml Zn 200.0 ug/ml
Point 1 = 0.5% HNO_3 Blank
Point 2 = 1-10,000 of Stock A
Point 3 = 1-1000 of Stock A
Point 4 = 1-100 Stock A

Group B calibration points are from a multielement standard (Stock B) containing the following...

Ca 1,000 ug/ml K 1,001 ug/ml Mg 500.0 ug/ml Na 1,000 ug/ml
Point 1 = 0.5% HNO_3 Blank
Point 2 = 1-1,000 of Stock B
Point 3 = 1-100 of Stock B
Point 4 = 1-10 Stock B

Group C calibration points are from a multielement standard (Stock C) containing the following...

Ag 100.0 ug/ml Al 600.8 ug/ml Mo 200.7 ug/ml
Point 1 = 0.5% HNO_3 Blank
Point 2 = 1-5,000 of Stock C
Point 3 = 1-1000 of Stock C
Point 4 = 1-100 Stock C

Group D calibration points are from a multielement standard (Stock D) containing the following...

As 199.8 ug/ml Pb 200.8 ug/ml Se 200.0 ug/ml
Point 1 = 0.5% HNO_3 Blank
Point 2 = 1-500 of Stock D
Point 3 = 1-200 of Stock D
Point 4 = 1-100 Stock D

Calibration Lines for metals under the Accustd Protocol are run as follows:

Boron Stock at 20 ug/ml
Point 1 = 0.5% HNO_3 Blank
Point 2 = 1-1000 of Stock
Point 3 = 1-100 of Stock
Point 4 = 1-10 Stock

Vanadium Stock at 20 ug/ml
Point 1 = 0.5% HNO_3 Blank
Point 2 = 1-1000 of Stock
Point 3 = 1-200 of Stock
Point 4 = 1-10 Stock

Titanium at 100 ug/ml and Strontium at 10 ug/ml
Point 1 = 0.5% HNO_3 Blank
Point 2 = 1-1000 of Stock
Point 3 = 1-100 of Stock
Point 4 = 1-10 Stock

Silica at 100 ug/ml and Tin at 40 ug/ml
Point 1 = 0.5% HNO_3 Blank
Point 2 = 1-1000 of Stock
Point 3 = 1-100 of Stock
Point 4 = 1-10 Stock

XI. Data Reduction:

- Metals in Groups A, C and D are generated as ug/L or ppb. Reported results must be in mg/L or ppm so all values for metals in groups A, C and D must be divided by 1000 before reporting.
- Metals in Group B are generated as mg/L or ppm so they are reported as is.
-Soil/sludge values are reported as mg/Kg dry weight. These values are calculated using the following equations:

$$\frac{\text{Conc. From Instrument(mg/L) x Digestion Volume(L)}}{\text{(Mass Digested(Kg) x Percent Solid(as a decimal)}} = \text{Sample Conc. Mg/Kg dry wt.}$$

XII. Data Recording/Archival:

-After a chain of custody is placed in the incoming box on the first floor, it is checked by an analyst who records any metal analysis onto the metals work log.
-The work log includes information such as Reference number, Order ID, Client, Due Date, if it's a RUSH, the type of digestion and the metals requested.
- A metals digestion log is kept to record when samples are digested
-A 3 ring binder is used to store raw data on the benchtop. A sticker is placed on the front page of the first data set of the day. The sticker records the date, analyst, standard numbers, and the date when all standards or reagents were made. The folder number listed on the top of the data print out is the date on which the samples were run.
- A standards log is kept to record when new stocks are made up.
-An instrument maintenance log is kept to record any maintenance or routine cleaning that may be done, such as when the water in the recirculator is changed or the pump lines are changed.
-Data and spreadsheets are backed up and stored on a labeled Zip drive.
-Hard copy raw data kept in hanging folders in the metals file cabinet.
-All raw data is archived annually in banker boxes and kept for a minimum of 5 years.

XIII. Troubleshooting:

For best results in starting up the ICP, turn on the following in this order:

Green power button on ICP
Open Argon tank completely, check to be sure there is sufficient gas left for the day (at least 1/4 tank) and that the pressure is at least 80 PSI
Turn on exhaust fan
Turn on water recirculator
Open up Leeman Labs DRE software

If the coolant won't turn on, shut down the software and try again. If this still doesn't work, shut off all ICP parts, reboot the computer and follow the steps above.
If the torch lights initially then immediately goes out, turn the power off by hitting the red button near the torch. Turn all other parts off and **quickly** turn off

and on the red switch on the left side of the ICP. This switch turns off the power supply to the capacitors. Therefore, let the ICP sit for 10-15min to insure the capacitors become fully recharged, then follow the steps above again.
Be sure to check the drain line from the pump to be sure the liquid is draining and not collecting in the spray chamber. If liquid does collect in the spray chamber, the torch will go out and not light correctly until the spray chamber is completely dry. If the line is not draining, slightly tightening or loosening the pump should help.
If little (< 10000) or no response is observed when peaking the X and Y axis for Mn, a new nebulizer is needed or reconditioning of the current one. To recondition the current nebulizer, place it in a 10% HNO_3 bath in a beaker in the sonicator for no more than 15 seconds and do not allow the nebulizer to touch the sides of beaker.
To extend the life of the pump lines, the following should be done at the end of each day:

25% HNO_3 should be run through for 5min
Next DI should be run through for 10min
Then the lines should be run dry
Release the tension on the sample lines

XIV. Revision History

February 2003
April 2004
October 2004
January 2005

XV. References

Method 6010B Inductively Coupled Plasma-AtomicEmission Spectrometry. EPA Test Methods for Evaluating Solid Waste Physical/ Chemical Methods, Version 2. EPA SW 846, December 1997 United States Environmental Protection Agency.

XVI. Method Modifications:

Appendix 1
Digestions/Sample Preparation

Dissolved Metals:

If the samples are not filtered in the field, they are filtered in the lab through 0.45µm filter paper using a magnetic filter and pump. The samples are then run "As is".

Total Metals (EPA 200.2):

-A 50ml aliquot of the well-mixed, acid preserved sample is poured off into labeled digestion cups. Next, 2ml of Conc. Nitric Acid and 1ml Conc. HCl are added. The cups are covered with plastic watchglasses and placed in the block digester for 8-9hrs at 95^0C.

-The samples are then brought back up to 50mls with DI and run on the ICP.

-A digestion blank and LFB are digested with the samples and are analyzed with all samples digested on that particular day.

-The date is written on the digestion cups to aid in tracking quality control.

Caution! When adding Nitric Acid and HCl to leachates, do so very slowly and in the fume hood. These samples may foam over and give off a choking gas.

Metals in Soil/Sludge (EPA Method 3050B):

Soils: 2-3 grams of the sample is placed in the digestion cups. 2.5 mls of 50% Nitric Acid is added. Sample is digested at 95^oC for 15 minutes. An additional 2.5 mls of Conc. Nitric Acid is added to each sample.

These samples only require 3-4hrs in the block digester at 95^0C. However, if they are being digested with other Total metals, they can be left in for 8-9hrs. If this is the case, add 30-40mls of DI to the samples so they won't dry up.

1 ml of DI and 1.5 mls of 30% Hydrogen Peroxide is then added and the samples are returned to the block digester at 95^0C until they begin to effervesce. They are allowed to cool and an additional 1 ml of 30% Hydrogen Peroxide is added. They are warmed again until they begin to effervesce and removed to cool. Then they are digested for an hour at 95^oC.

To samples being run on the ICP 5mls of conc. HCl is added and digested for 1 hour.

The samples are then brought back up to 50mls with DI

A 2.0 um Teflon fiber filter is then used to be sure no large particulate matter is present that would block the lines of the ICP.

Sludges: Sludges are digested just like Soils, except that an initial weight of 20-30 grams of sample is used.

Metals in Air (NIOSH 7300):

Make ashing acid **IN HOOD WITH ALL PPE** - In 200ml Volumetric Flask add 40mls Conc. Perchloric Acid ($HClO_4$) bring to the mark with Conc. Nitric Acid (HNO_3)

Make Dilution Acid - 50mls Ashing Acid to 600mls DI, dilute to 1L

Place Cellulose filters (samples, QC's, Blanks etc.) in 50ml Digestion cups.

1. Add 5mls Ashing acid, cover with watchglass and let sit for 30min at room temperature.
2. Heat in digestion block at 95^oC until 0.5ml remains.
3. Add 2mls Ashing Acid and repeat step 2 until solution is clear.
4. Remove watchglass and rinse residue into digestion cup with DI. Let heat until 0.5mls or less remains. Digest 3-4 hours max.
5. Bring to 50mls with dilution acid.
6. Separate calibration lines are run using the dilution acid matrix.

Table B

Calibration and Standard Concentrations

The highest point in each calibration line is also the point at which Leeman Lab DRE software updates the slope for each analyte.

ACD SLOPE

There is an ACD slope solution consisting of the metals in groups A, C, and D. Use the multielement stock standards for A, C and D as follows:
In a 200ml volumetric flask add 2mls of each stock standard and fill to the mark with 0.5% HNO_3.

B SLOPE

A separate slope solution is made for the metals in Group B.
In a 200ml volumetric flask add 20mls of stock standard B and fill to the mark with 0.5% HNO_3.

INTERFERENCE CHECK

This is analyzed at the beginning of each analytical day. Refer to the “working standards log” in the metals laboratory for specific concentrations.

CHECK STANDARD 2

This is a line check for each analyte and is run at the beginning, end and every 10 samples.
In a 200ml volumetric flask add 1ml each of stock standards A, C and D and 5mls of stock standard B. and fill to the mark with 0.5% HNO_3.

CHECK STANDARD 3

This is a line check for each analyte in the Accustd protocol, and is run at the beginning, end and every 10 samples. Refer to the “working standards log” in the metals laboratory for specific concentrations.

TABLE C ICP Reporting Limits

Element	ppb
Ag	10
Al	60
Ba	20
Be	2
B	20
Ca	100
Cd	3
Co	10
Cr	10
Cu	10
Fe	10
K	500
Mg	100
Mn	5
Mo	20
Na	200
Ni	20
Sb	60
Sn	40
Si	100
V	20
Zn	20
(Pb)	100
(As)	100
(Se)	100
(Au)	20
(Bi)	250

XVI. SOP Attestation

I acknowledge that I have read this Standard Operating Procedure and agree to adhere to the Safety and Quality Control policies outlined within.

Name	Date

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MERCURY IN SOLID OR SEMISOLID WASTE

Method SW 7471A

Standard Operating Procedure

REVISED: September 15, 2006

Revision #: 4

Technical Director: _____Date :_____
Harry B. Locker Ph.D.

QA Officer: _____Date :_____
Mark L. Fausel

Document Control Copy on Buff colored paper. Any other color is not official version

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I. Overview:

This method employs the Varian VGA-76 Vapor Generation Assembly, combined with the SpectrAA 640Z Atomic Absorption Spectrometer, for determination of mercury in soils, sediments, bottom deposits and sludges.

Prior to analysis, the solid or semi-solid samples must be prepared according to the procedures discussed in this method.

This cold-vapor atomic absorption method is based on the absorption of radiation at the 253.7 nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of the atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

II. Safety:

This method involves the handling of the chemicals outlined below. Each of these chemicals may have specific associated hazards. Analysts must read and become familiar with all elements of each chemical's MSDS. Make special awareness of the symptoms of exposure and potential routes of entry. Analysts must notify the Safety Officer immediately if any symptoms of exposure are suspected.

PPE must be worn at all times in designated work areas. All applicable engineering controls, such as fume hoods or splash shields, must be used when performing analyses. To ensure the safety of all laboratory personnel; work areas must be kept neat and organized, equipment and bench-tops must be cleaned and sterilized, if necessary, at the completion of each task, all containers must be properly labeled as to contents and hazard ratings and spills cleaned immediately and completely.

III. Waste Disposal:

Samples that do not contain mercury may be flushed down the drain with copious amounts of water. Samples containing mercury, as well as all standards and QC samples, must be disposed of in the Hazardous Waste drum. Expired hollow cathode lamps may not be placed in the trash; they are considered hazardous waste since they contain the target element(s). Lamps should be disposed of in an appropriate waste stream or returned to the vendor for recycling.

IV. <u>Sample Collection, Preservation, and Storage:</u>

A. Collection:
   Samples are collected in plastic and glass bottles of various sizes.
B. Labeling:
   All samples are assigned a sample number, plus a unique identifier suffix to indicate preservative and bottle type.

C. Preservation:
Non-aqueous samples, or those known to react violently with acid preservation, are stored in the inorganics refrigerator below 4°C and analyzed as soon as possible.

D. Storage:
Samples are stored in the inorganic fridge.

V. Equipment and Supplies:

Digestion Tubes with screw caps
Block digester capable of maintaining 95°C +/- 5°C
Volumetric Flasks, Various Sizes Class A
Mercury Hollow Cathode Lamp
Varian SpectrAA 640Z Spectrophotometer
Varian Vapor Generation Accessory (VGA-76)
Gas/Liquid Separator
Absorption Cell
Flouroelastomer Tubing
Nitrogen, Compressed Gas

VI. Reagents and Standards:

Reductant #1 - 12% W/V Sodium Chloride and 12% W/V Hydroxylamine hydrochloride in DI. Ex. Dilute to 30g of each compound to 250 mL
Reductant #2 - 10% Stannous Chloride in 20% HCl. Ex. 20g→200mls
Nitric Acid, concentrated, Trace Metal Grade
Hydrochloric, concentrated, Trace Metal Grade
Potassium Permanganate, 5% W/V ex. 50g→1L
Mercury Stock Standard

NOTE: All incoming reagents, standards, dry chemicals and acids shall be marked with the date of receipt, date of opening, and date of expiration. Dry chemicals shall be used within 20 years, and concentrated acids shall be used within 10 years of the date of receipt or before the manufacturer's expiration date, which ever is sooner.

VII. Interferences:

The vapor generation technique demands a high standard of care: scrupulous cleanliness is essential in all laboratory procedures; standards and samples must be meticulously prepared and carefully handled; and strict precautions must be taken to avoid contamination of the apparatus.

VIII. Quality Control

A. (See Table A in the "SOP" SOP for more detailed definitions of the following)
1. Demonstration of Capability (DOC). Each new analyst performs a set of four mid-range LFBs. The LFBs should be prepared as one sample, then split into four

aliquots. The percent standard deviation and average percent recovery are determined for each analyte. Each must be less than 20% to ensure proper capability of the analyst for that method. This process is repeated annually for each metals analyst.

2. Method Detection Limit (MDL).
   a. An MDL is run annually for each element. The final calculated MDL value must be at least ½ the reporting limit and the MCL. MDLs should be prepared in a manner identical to that of sample analysis.
   b. Seven to ten QCs are made at at concentration 2-5 times highger than the expected MDL.
   c. The MDL is calculated by taking the standard deviation of the values and multiplying by the appropriate number from the chart from the Code of Federal Fregulations Appendix B to part 136 of Chapter 1 (7 is 3.143, 8 is 2.998, 9 is 2.896and 10 is 2.821).

3. Calibration Check/Verification (or Instrument Performance Check – IPC). The concentrations of the calibration verification shall be varied within the established calibration range.
4. Laboratory Reagent Blank (LRB).
5. Laboratory Fortified Blank (LFB or Independent QCS)
6. For all determinations the laboratory must analyze a calibration blank and line check (IPC) at the start of the sample run, after every tenth sample (or more frequently, if required) and at the end of the sample run.
7. The percent recovery for IPC's must be +/-10%.
8. The calibration blanks can not be greater than 1/10 of the measured concentration of any sample in the associated sample batch; or exceed the concentration present in the samples or be greater than 1/10 of the regulatory limit.
9. Percent Recovery for the LFB must be +/-15%.
10. Duplicate samples are run every batch or every 10 samples
11. LFM samples are run every 10 samples or when results are questionable. Percent recovery for LFM's must be +/-30%.
12. LFM's are acceptable as Independent QC's.
13. If data does not meet the criteria outlined above, it may be used, but MUST be noted on the final client report.

IX. Analytical Procedure:

Digestion:

1) Turn on block digester and set at 95°C.
2) Record sample reference number on the Hg page of the metals digestion log. Record all pertinent information concerning reagents and standards.
3) Prepare a blank and 1, 2, 4, 6, and 8 ppb standards from a 1 ppm Mercury solution - each at a volume of 30 mLs with **3% Nitric acid solution** (30ul of 1.0ppm Hg into 30mls DI equals 1.0ppb. Prepare QC's at 5 ppb (150ul of 1.0ppm QC into 30mls DI) in digestion tubes. Samples may also be run against the water line since the absorbances have proven to be the same for both lines.
4) A duplicate (LD) and spike (LFM) should be run with each project or C.O.C. with a minimum of one dup and spike per digestion. The spike target may be between 3-5 ppb.
5) Using the soils spreadsheet calculate the amount of sample needed to yeild a desireable detection limit. Weigh out that amount of sample (in three separate weights to assure a representative sample) into a digestion tube. Record the mass of each sample used on the Hg page of the digestion log. Add **30 mLs of 3% Nitric Acid** solution to each soil or sludge sample.
6) After samples and standards are prepared, add **0.5 mL concentrated Nitric Acid and 2.0 mLs concentrated Hydrochloric Acid** to each tube. Mix thoroughly. Heat 10 min in 95^{0}C block.
7) Add **3.0 mL Potassium Permanganate** to each tube. Mix thoroughly. Allow to sit for 15 minutes. If purple color does not persist then add an additional 3.0 mLs Potassium Permanganate to each tube.
8) Place in block for 30 minutes at 95^{0}C.
9) Remove bottles from block and cool for 20 min. Add **3.0 mls Reductant #1** to each tube, and swirl until all purple particles are gone.
10) Allow samples to cool to room temperature, filter, and dilute (using digested blank) before analysis.

Analysis:

1) Disconnect magnet BEFORE power is applied to the Spec. Remove the furnace workhead and autosampler. Turn on Spec and software. Install VGA on to front of Spec. Attach liquid-gas separation chamber. Hook drain tube to bottom right side of liquid-gas separator, connect line from reaction coil to stem on left side, and inert gas supply line to bottom left (see VGA-76 instruction manual). Allow Hg lamp to warm-up **at least one hour** before use.
2) Optimize lamp signal so that the Hg beam passes through the absorption cell by adjusting the cell bracket. The lamp wavelength should be 253.7 nm.
3) Insert black fluoroelastomer tube into the left stem of the absorption cell making sure that a tight seal is formed. Insert the other end to the top right stem on the liquid-gas separator. Install absorption cell exit hose with extra 5% permanganate solution to trap Hg gas as it exits the cell.
4) Place reagent tubes around pump rollers. Check for cleanliness. If tubes are dirty or worn, replace before analysis.
5) Adjust tension screw on front bed until water just begins to pump through the tubes

6) Leave tubes in DI for about 5 minutes for rates to stabilize.
7) Check nitrogen flow with a flowmeter. Flow should be 1 L/min. The pressure should be approximately 45 psi.
8) Determine flow rates for each tube by measuring the volume of DI pumped up over a given time. The flow rate for the sample tube should be about 8 mL/min. The flow rates on the other two tubes should be about 1 ml/min. The tension screws should be adjusted to achieve these rates.
9) Place sample, DI, and Stannous Chloride (reductant 2) tubes in appropriate containers.
10) Allow system to stabilize (check by reading a cal zero standard until signal stabilizes).
11) Begin Autorun sequence after preparing Method, Sequence, and Instrument pages in the software.
12) Insure that there is not an excessive amount of foam in the gas/liquid separator during analysis.
13) When analysis is complete, place all three tubes back in DI for five minutes and then air dry for 5 minutes.
14) Shut off power to VGA and remove tubes from pump rollers.
15) Remove absorption cell and gas/liquid separator. Sonicate the gas/liquid separator and the 2 gray fluoroelastomer tubes in 10% HNO_3 for 5-10 minutes. Rinse well with DI and air dry.
16) Store the VGA away.

X. <u>Calibration</u>:

A. Calibration is performed by analyzing a series of digested standards from zero to 8ppb. The Varian software will then prepare a calibration curve by plotting instrument response versus standard concentration. The calibration line may be digested or undigested. For an undigested line, follow the procedure listed in IX and omit step #9.
B. A digested calibration line is often stable for several weeks.
C. For all determinations the laboratory must analyze a calibration blank and Line Check at the start of the sample run, after every tenth sample and at the end of the sample run. The Line Checks must have a recovery of +/- 10%
D. An LFB must be analyzed at the start of each sample run. Percent recovery for LFBs must be +-15%
E. Duplicate samples are run every batch or every 10 samples
F. LFM samples are run every 10 samples or when results are questionable. The percent recovery for LFM's should be +/- 30%
G. The instrument operating conditions are as follows:

Instrument Operating Conditions for Mercury
General

Method 6

Instrument Mode	Absorbance
Instrument Type	Vapor
Calibration Mode	Concentration
Calibration Algorithm	Linear NZ
Measurement Mode	Integration
Time Measurement	5.0 Sec
Read Delay	40 Sec
Sampling Mode	Manual
Lamp Position	Varied
Lamp Current	5 mA
Slit Width	0.5
Slit Height	Normal
Wavelength	253.7
EHT	320-360
Minimum Reading	0.000
Replicates All	2
Background Correction	None
Standard 1 concentration	1.000
Standard 2 concentration	2.000
Standard 3 concentration	4.000
Standard 4 concentration	6.000
Standard 5 concentration	8.000
Smoothing	5 Point
Expansion Factor	1.0
QC Protocol	On
Control Rate	10
Time Limit	120
Initial Concentration	5.000
Continuing Concentration	5.000
Initial Recovery limts	90-110%
Continuing Recovery Limits	90-110%
Action Initial	Flag and Continue
Action Continuing	Retry Flag and Continue
Concentration	0.0000
Action	Flag and Continue

QCP Parameters

QC Standard	
Concentration	5.000
Minimum Recovery Limit	40
Sample % of Spike	50
Continuing Recovery Limits	90 % to 110 %
Action	Flag and Continue
Action	Flag and Continue
QC Blank	
Concentration	1.0000

Volume	20
Action	Flag and Continue
Matrix Spike	
Concentration	5.0000
Recovery Limit	70 % to 130 %
Action	Flag and Continue
Duplicates	
For Conc >	3.0000
RPD limit	20.000 %
Or Conc Limits	-0.5000 to 0.5000
Action	Flag and Continue
Other Limits	
Replicate %RSD Limit	20.0000
Correlation Coefficient	0.9950
Required Detection Limit	1.0000
Instrument Detection Limit	0.923

XI. Data Reduction:

Insure that any dilutions are factored into the final result.
Solid samples are reported as mg/kg, dry wt. The calculations may be performed using an Excel spreadsheet. The calculation is as follows:

$$\frac{\text{(Conc. in PPM)(Vol. In L)(1000)}}{\text{(\% Solid)(Mass in G)}} = \text{Mg/Kg, dry wt.}$$

XII. Data Recording:

1. All analyses are recorded on the Mercury page of the MetalsDigestion Log Each log sheet should include the sample reference number, analyst's name, and concentrations for standards, LFB's, and LFM's
2. Record final data in the LIMS databank.
3. Raw data is stored in the binder labeled "AA Metals" located in the Inorganics laboratory.
4. For long-term storage, data is stored in a banker's box labeled "AA Raw Data".
5. Data from the hard drive C is backed up onto the partitioned D drive

XIII. Instrument Maintenance:

A. Refer to instruction manual for preventative maintenance procedures for the VGA-76.
B. Replace sample and reagent tubes on the VGA-76 at least monthly, or when they appear discolored or worn.
C. Quarterly check gas supply for the VGA-76 for leaks. Use a flowmeter to measure incoming pressure and compare with specifications listed in the operation manual.

XIV. Troubleshooting:

A. If condensation is present in the flow cell, install and turn on a heat lamp over spec. to facilitate approximately a 10°C rise in the ambient temperature in the cell.
B. Filter soil samples to prevent a clog in the sample line, using a 2.0 um Teflon fiber filter.
C. Rinse sample line thoroughly with DI between high samples
D. There should be gentle bubbling in the gas-liquid separator. If not, disconnect the inlet line to the absorption cell, allow the separator chamber to fill up with DI, then re-attach inlet line. Still no bubbling, remove inlet line and clean out with compressed air.
E. If contamination is suspected compare the EHT voltage on the optimization page with the original value from the beginning of the run. An elevated voltage indicates that there is Hg in the system.

XV. References

Method 7471A: Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique) Revision 1,September 1994, EPA Test Methods for Evaluating Solid Waste Physical/ Chemical Methods, Version 2. EPA SW 846, December 1997 United States Environmental Protection Agency.

Electronic Code of Federal Regulations, 40 CFR – Chapter 1 – Part 136 – Appendix B. : Definition and Procedure for the Determination of the Method Detection Limit – Revision 1.11.

XVI. Revision History

May 14, 2003
March 26, 2004
April 17, 2006

XVII. Method Modifications/Analyst Notes:

XVIII. SOP Attestation

I acknowledge that I have read this Standard Operating Procedure and agree to adhere to the Safety and Quality Control policies outlined within.

Name	Date

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Experience is the solution
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Total Organic Carbon - Laboratory QC Limits

Total Organic Carbon - Water

Analyte	LCS	Duplicate	Spike
Total Organic Carbon	84.9-120 %	20.7 %	79.8-121 %

Total Organic Carbon - Soil

Analyte	LCS	Duplicate	Spike
Total Organic Carbon	77.9-119 %	20 %	75-125 %

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Total Organic Carbon in Sediment

Application: This method describes protocols for the determination of organic carbon in sediment and soils. Data are reported in mg/kg on a dry weight basis. Wet combustion methods are not considered to be equivalent to the pyrolytic method.

Interferences: Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias.

Detection Limit: A reporting limit of 100 ug/g is used for this test.

Method Summary: Inorganic carbon from carbonates and bicarbonates is removed by acid treatment. The organic compounds are decomposed by pyrolysis in the presence of oxygen or air. The carbon dioxide that is formed is determined by direct non-dispersive infrared detection. Water content is determined on a separate portion of sediment.

Preservation & Storage: Sample should be collected unpreserved and kept at 4 °C until analysis. Samples should be analyzed within 14 days from collection.

Definitions: See General Laboratory Definitions Page

Safety: No special safety requirements are necessary for this test method. General laboratory safety precautions should be followed. Treat all samples as potentially hazardous.

Reference Method: Lloyd Kahn, EPA Region 2

Apparatus:

1. Tekmar TOC analyzer – Phoenix 8000 TM
2. Nitrogen Tank with regulator
3. 1000 uL syringe, 250 uL syringe
4. Pump Tubing
5. Volumetric Flasks
6. Analytical Balance

Reagents:

1. Reagent Water: The total organic carbon value of the reagent water should be less than 1 mg/L.
2. Potassium hydrogen phthalate, stock solution (2000 mg/L Carbon): Dissolve 4.2508 g of potassium hydrogen phthalate (Primary Standard Grade) in reagent water and dilute to 1 liter. Preserve with Phosphoric acid or Sulfuric acid to pH > 2. This solution is good for 6 months.
3. Potassium hydrogen phthalate, working solution (400 mg/L Carbon): Dilute 20 mL of the stock solution to 100 mL in a volumetric flask. Prepare monthly
4. Acidified Persulfate Reagent: Place 100 mL of reagent water in a container. Add 5 g of potassium persulfate. Add 3 mL of concentrated (85%) phosphoric acid.

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Procedure:

1. Make sure that the reactor vessel is filled with the persulfate solution. Fill vessel to just below the ground glass joint.
2. Assemble the pump assembly is not already done.
3. Turn on the main power, reactor power, UV Lamp and pump.
4. Turn on the Nitrogen gas to 30 psi.
5. Allow 10 minutes for warm-up.
6. Select range. Usual range 40 uL injection.
7. Inject 40 uL of the 2000 mg/L stock TOC solution for calibration.
8. The sample received is dried in an oven to remove moisture.
9. After drying, a portion of the sample is then weighed into the sample boat and placed in the analyzer for analysis. Heterogeneous samples are mixed as thoroughly as possible. The amount used is much smaller than the total sample supplied. Generally the sample weight used for analysis is approximately 2.0-10.0 mg, depending on the TOC content. The higher the TOC content, the lower the sample weight used for analysis.
10. Due to these factors, a percent difference of greater than twenty percent is quite common for this type of matrix. The samples are analyzed in duplicate and the average result reported.
11. In the analyzer, the sample is heated in a resistance furnace under oxygen, the interfering gases removed by a sparger/scrubber system and the resultant Carbon Dioxide is measured using a non-dispersive infrared (IR) detector.
12. When analysis is complete turn off the UV light, pump and Nitrogen gas. Leave IR unit on. Release the pump assembly.

The calculations for this test method are as follows:

Sample calculations for TOC in Soil/Sediment

Instrument calibrated with a 2000 mg/L aqueous standard. Instrument print-out in mg/L. The values in mg/L are equivalent to ug/mL. The calculation for the sample concentration in ug/g is given by the following:

Concentration of TOC in sample:

$$\text{TOC ug/g} = \frac{\{\text{sample reading from instrument (ug/mL)}\}\{\text{volume of syringe(mL)}\}}{\{\text{weight of sample used for analysis(g)}\}}$$

Example Calculation Replicate 1:

Weight of sample used: 0.0015 g

Capacity of syringe used for calibration: 0.040 mL

Reading from instrument: 2960 ug/mL

Example Calculation Replicate 2:

Weight of sample used: 0.0012 g

Capacity of syringe used for calibration: 0.040 mL

Reading from instrument: 2932 ug/mL

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Replicate 1

$$\text{TOC ug/g} = \frac{(2960)(0.040)}{0.0015}$$

TOC ug/g = 78,933

TOC % = 7.89

Replicate 2

$$\text{TOC ug/g} = \frac{(2932)(0.040)}{0.0012}$$

TOC ug/g = 97,733

TOC % = 9.77

The average of two sample portions is taken and this average is reported on the final report as 8.8 %.

Required Quality Control (see current acceptable limits):

LCS: Once each analytical batch or every 20 samples.

Spike: Once every 20 samples.

Duplicate: All samples are analyzed in duplicate. Run an analytical duplicate once every 20 samples.

QC Acceptance: Quality control acceptance criteria is generated by the QA Manager and posted in the LIMS system and on hard copy in each laboratory.

Corrective Actions: If the continuing calibration check or Laboratory Control Sample is not within acceptable criteria, the sample must be reanalyzed. If the continuing calibration check or Laboratory Control Sample has a high bias and the samples are below the detectable level the sample results may be reported without any flags. If the duplicate is out of specified limits re-run the duplicate on a different sample to verify whether the problem is with one particular sample or the method. The data should be flagged and the client notified of the quality control outside of specific limits. This may be due to the sample matrix.

Method Performance: See test method for specific method performance. Quality Control Charts are maintained on the LIMS system for all analytes.

Pollution Prevention: Reduce the amount of chemicals required when possible for this method in order to reduce potential wastes.

Waste Management: See Waste Disposal SOP for details on disposal of potential wastes from this method.

Method Validation: See published test method for method validation, additional flowcharts and tables.

Reviewed by QA Manager: Cathy Hess

Date Reviewed: 7/18/06

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Total Organic Carbon

Application: This method covers the determination of total organic carbon in drinking water and other waters. This method is applicable only to the carbonaceous matter which is either soluble or has a particle size of 0.2 mm or less. The applicable range is from approximately 1 mg/L to 200 mg/L. Higher concentrations may be determined by sample dilution.

Interferences: If a sample is homogenized to reduce the size of the particulate matter, the homogenizing may cause loss of purgeable organic carbon, thus yielding erroneously low results.

Detection Limit: A reporting limit of 1 mg/L is used for this test. Actual MDL is on file with the Quality Assurance Manager.

Method Summary: The sample is first sparged with nitrogen to remove any CO_2 that may be present. The sample is injected into a quartz ultraviolet reaction coil where the organic carbon is subjected to intense ultraviolet illumination in the presence of the acidified persulfate reagent. The organic carbon is converted to CO_2 and a nitrogen purge transfers the CO_2 to the reduction system and into the detector. The signal is integrated and displayed as the concentration of total organic carbon.

Preservation & Storage: Sample should collected preserved with Phosphoric acid to a pH of < 2 and kept at 4 °C until analysis. Samples should be analyzed within 28 days from collection.

Definitions: See General Laboratory Definitions Page

Safety: No special safety requirements are necessary for this test method. General laboratory safety precautions should be followed. Treat all samples as potentially hazardous.

Reference Method: SM 19th Edition Method 5310C

Apparatus:

1. Tekmar TOC analyzer – Phoenix 8000 TM
2. STS 8000 Autosampler
3. Nitrogen Tank with regulator
4. Volumetric Flasks
5. Analytical Balance

Adirondack Environmental Services

Reagents:

Organic Carbon Reagents

1. Reagent Water: Use the carbon tap DI water in volatiles. The total organic carbon value of the reagent water should be less than 1 mg/L.
2. Potassium hydrogen phthalate, stock solution (2000 mg/L Carbon): Dissolve 4.2508 g of dried potassium hydrogen phthalate (Primary Standard Grade) in reagent water and dilute to 1 liter. Preserve with Phosphoric acid to pH > 2. This solution is good for 6 months.
3. Potassium hydrogen phthalate, working solution (400 mg/L Carbon): Dilute 20 mL of the stock solution to 100 mL in a volumetric flask. Prepare monthly.
4. Sodium Bicarbonate
5. Concentrated Phosphoric acid (85 %).
6. Acidified Persulfate Reagent: Place 213 mL of reagent water in a container. Add 25 g of potassium persulfate. Add 9 mL of concentrated (85%) phosphoric acid.
7. 21 % Acid reagent – Add 37 mL of concentrated (85%) phosphoric acid to 188 mL of reagent water.

Inorganic Carbon Standard

8. Carbonate-bicarbonate, stock solution, 1000 mg carbon/liter: Place 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate in a 100 mL volumetric flask. Dissolve with reagent water and dilute to the mark. Do not acidify.

Procedure:

1. Fill the gas-liquid separator with carbon tap De-ionized water with 0.25 mL H_3PO_4.Make sure that this is filled before each run.
2. Turn on the main power, reactor power and UV Lamp.
3. Turn on the Nitrogen gas to 30-35 psi.
4. Allow 30 minutes for warm-up.
5. Select curve. Usual range is 20-200 mg/L.
6. Set up autosampler and run log.
7. The samples are automatically sparged and the instrument performs analysis in triplicate for each sample.
8. When analysis is complete turn off the UV light and Nitrogen gas. Leave IR unit on. Instrument should shut down on its own.

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Instrument Set up

-Refer to the Preventative Maintenance Chart (table 6-1) for the daily,weekly and monthly maintenance

-Turn gas on the day before starting a run-will decrease warm up time

-gas on 30-35 PSI (never >35)

-If main screen is not up, click on the Phoenix Toc Talk 3.0 icon

-In SET UP MENU choose INSTRUMENT - click on the circle to the left of READY, the ready light, and the UV lamp should turn on.

-Flow meter to UV should read 200 +/- 10 (the from detector flow meter is not working so we need to check the outflow manually), if not adjust PSI. Click OK

-click on RUN

-Adjust size of back screen to see NDIR MV - should be between 5-10 before beginning run
Should be OK after 30-60 minutes.

To set up a run

-click on SAMPLE SET UP. The previous sample setup will open.

-To open a sample setup template - click on FILE, then OPEN, scroll down to find setup or type name in text box. ("Basic")

- Use Basic or Basic2(rinse vials in between samples)

samples-3 repetitions
rinses(if using Basic2 setup) - 1 rep
CCV,CCB - every 10
LCS - after ICB then every 20 samples
dup - every 20 samples
spk - every 20 samples

cleaning-5 reps (position 1001)
reagent blank - 5 reps (position 1001)

*CLP needs CRDL after LCS

ICV,CCV 100 or 50 mg/L

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*cleaning-do not exceed 5 reps water may get too hot for multiport valve and may melt teflon rotor
-Tech support says that raw data after 5 reps should be 30,000. It usually will go down to ~60,000.

-To Change Text In Sample ID Cells

Click on cell and begin typing
*Pressing delete will delete entire row. Press delete only when cursor is flashing inside cell

-RESET STATUS COLUMN
- -go to EDIT menu
- -choose RESET STATUS
- -If you have filled all rows
  - -choose ALL ROWS then click OK
- -If you have not filled all rows
  - -choose RANGE
  - -fill in range selection using the row #'s in the 1st column
  - *1st column refers to setup rows, the 2nd column refers to vial position numbers
  - ANY ROW WITH SKIP STATUS WILL NOT BE ANALYZED

OR

You can delete any rows that you do not want

-If you need to add rows-go to the last row and press the down arrow key. You may have to adjust the other columns

-Save Your Setup As A Different Name
- -go to FILE menu
- -choose SAVE AS
- -type in name you want for setup(ex.092905), click OK
- -click SAVE/USE to use setup for the run

-Start Run
- -check NDIR MV on bottom of screen, should be between 5-10
- -click on START

Changing Sample Setup While Run Is In Progress

-click on SAMPLE SETUP and choose OK at the caution menu

-If you have the setup screen open while the run is progress you will not see the status column change. So, if a sample has started and finished while you had the setup screen open the status of that sample will still appear as READY. If you click on the Save/Use button the autosampler will go back to that vial (the autosampler will always go to the uppermost READY).

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-To avoid this from happening change the status of anything that was running or has started and finished while you had the setup screen open or anything that is currently running to SKIP.

-After you've checked the status column, click on the SAVE/USE button.

-If you just opened the setup screen to look at it and you are not changing anything, then click on EXIT to close the screen

Viewing And Printing Results

-The Analysis Results screen shows only a limited number of results. To see results from current or most recent run - select RESULTS from the back screen, then MULTIPLE ANALYSIS.

To See Results From Other Runs

-from Multiple Analysis screen, choose FILE
-then choose OPEN RESULTS
-find file name - 09291121.prn means that this run was started on September 29th at 11:21.
-choose the filename you want then click OK

* BEFORE OPENING FILE, CLEAR CURRENT SCREEN IF IT DOES NOT CONTAIN INFO THAT YOU WANT PRINTED (choose CLEAR under the FILE menu)

* If you stopped a run then restarted it late, it will be saved under 2 filenames.

To Print The Multiple Analysis Screen

-In Multiple Analysis screen - go to FILE, then PRINT DETAILED REPORT
-choose yes for margins

Calculations:

Read the concentration of TOC directly from the instrument print out. Multiply readout by any dilutions performed.

Required Calibration criteria:

Use linear regression for calculation of the curve. The calibration curve is analyzed using a blank and standards at concentrations of 1 mg/L, 10 mg/L, 50 mg/L, 100 mg/L and 200 mg/L. The correlation coefficient must be 0.997 or greater.

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Required Quality Control (see current acceptable limits):

ICV/CCV: Once every 10 samples.

ICB/CCB: Once every 10 samples immediately following the ICV/CCV.

CRDL: Once each analytical batch or every 20 samples.

LCS: Once each analytical batch or every 20 samples.

Spike: Once every 20 samples.

Duplicate: Once every 20 samples.

QC Acceptance: Quality control acceptance criteria is generated by the QA Manager and posted in the LIMS system and on hard copy in each laboratory.

Corrective Actions: If the continuing calibration check or Laboratory Control Sample is not within acceptable criteria, the sample must be reanalyzed. If the continuing calibration check or Laboratory Control Sample has a high bias and the samples are below the detectable level the sample results may be reported without any flags. If the duplicate is out of specified limits re-run the duplicate on a different sample to verify whether the problem is with one particular sample or the method. The data should be flagged and the client notified of the quality control outside of specific limits. This may be due to the sample matrix.

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Pollution Prevention: Reduce the amount of chemicals required when possible for this method in order to reduce potential wastes.

Waste Management: See Waste Disposal SOP for details on disposal of potential wastes from this method.

Method Validation: See published test method for method validation, additional flowcharts and tables.

Reviewed by QA Manager: _____

Date Reviewed: 7/18/06

APPENDIX H
ENDYNE 2007 QUALITY CONTROL DATA

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Endyne, Inc.
160 James Brown Drive
Williston Vermont

Control Limits 2006 EPA 8260B/624 - Ind. Standard as Percent Recovery - Solid Samples

Parameter	PRG mg/kg	Reporting Limit ug/Kg**	Method Detection Limit ug/Kg	MEAN Percent Recovery	Standard Deviation	Upper Warning Limit %	Lower Warning Limit %	Upper Control Limit %	Lower Control Limit %	Duplicate Difference 100ppb D/MS
2) Dichlorodifluoromethane	9.4E+04	50.0	3.37	92.7	14.5	121.8	63.7	136.3	49.1	30.2
3) Chloromethane	4.7E+04	30.0	4.64	96.8	13.7	124.2	69.3	138.0	55.5	30.4
4) Vinyl chloride	7.90E-02	20.0	5.04	99.6	14.6	128.8	70.5	143.3	55.9	33.7
5) Bromomethane	3.9E+03	50.0	12.5	102.4	12.3	126.9	77.8	139.2	65.6	33.2
6) Chloroethane	3.0E+03	50.0	5.62	103.0	13.8	130.5	75.5	144.2	61.7	31.5
7) Trichlorofluoromethane	3.9E+05	20.0	4.11	98.2	13.2	124.5	71.9	137.7	58.7	30.9
8) Diethyl ether	NA	50.0	4.70	103.3	14.0	131.4	75.3	145.4	61.3	29.3
9) 1,1-Dichloroethene	1.2E+05	10.0	2.83	101.3	13.1	127.4	75.2	140.4	62.1	32.5
10) Acetone	1.4E+07	100.0	19.8	105.6	14.0	133.5	77.7	147.5	63.7	48.0
11) Carbon disulfide	3.6E+05	50.0	6.36	100.1	14.1	128.3	71.9	142.4	57.8	31.3
12) Methylene chloride	9.1E+03	50.0	8.04	105.1	13.9	132.9	77.4	146.7	63.5	31.8
13) MTBE	3.2E+04	20.0	4.93	99.6	12.2	123.9	75.2	136.1	63.1	33.1
14) trans-1,2-Dichloroethane	NA	10.0	4.22	103.0	12.7	128.4	77.6	141.2	64.8	30.2
15) 1,1-Dichloroethane	5.1E+05	10.0	4.95	103.3	12.4	128.2	78.4	140.6	65.9	32.0
16) 2-Butanone	NA	100.0	7.39	105.3	14.8	134.9	75.7	149.7	60.9	55.4
17) 2,2-Dichloropropane	NA	10.0	3.32	98.0	12.3	122.6	73.4	134.9	61.1	29.3
18) cis-1,2-Dichloroethene	4.3E+04	10.0	3.85	100.6	10.7	122.0	79.3	132.6	68.6	23.8
19) Bromochloromethane	NA	20.0	2.95	101.0	11.9	124.7	77.3	136.6	65.4	24.7
20) Chloroform	2.2E+02	10.0	3.36	99.1	9.3	117.6	80.6	126.9	71.3	25.6
21) Tetrahydrofuran	9.4E+03	100.0	7.11	99.6	13.0	125.7	73.6	138.7	60.5	29.4
22) Dibromofluoromethane S1	surrogate	surrogate	surrogate	98.6	7.6	113.7	83.4	121.3	75.9	9.47
23) 1,1,1-Trichloroethane	1.2E+06	10.0	2.19	97.5	10.7	118.9	76.1	129.6	65.4	29.7
24) Carbon tetrachloride	2.5E+02	10.0	3.29	95.5	11.5	118.4	72.5	129.8	61.1	32.9
25) 1,1-Dichloropropene	NA	10.0	2.49	99.2	11.0	121.1	77.2	132.0	66.3	28.9
26) Benzene	6.4E+02	10.0	4.11	100.3	11.2	122.7	77.9	134.0	66.7	24.2

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Parameter	PRG mg/kg	Reporting Limit ug/Kg**	Method Detection Limit ug/Kg	MEAN Percent Recovery	Standard Deviation	Upper Warning Limit %	Lower Warning Limit %	Upper Control Limit %	Lower Control Limit %	Duplicate Difference 100ppb D/MS
27) 1,2-Dichloroethane	2.8E+02	10.0	3.78	98.1	9.3	116.7	79.4	126.1	70.1	23.6
29) Trichloroethene	5.3E+01	10.0	3.50	99.6	11.5	122.5	76.7	134.0	65.3	31.4
30) 1,2-Dichloropropane	3.4E+02	10.0	3.22	101.4	10.8	123.0	79.7	133.9	68.9	23.2
31) Dibromomethane	NA	20.0	3.90	97.4	8.8	115.0	79.9	123.7	71.2	22.8
32) Bromodichloromethane	8.2E+02	10.0	7.32	97.6	9.8	117.2	77.9	127.1	68.1	23.6
33) 2-Chloroethylvinylether	NA	200.0	3.33	97.7	15.8	129.4	66.0	145.2	50.2	40.3
34) cis-1,3-Dichloropropene	7.8E+02	10.0	5.30	98.5	8.9	116.3	80.7	125.2	71.8	22.0
35) MIBK	NA	100.0	3.29	97.4	13.6	124.6	70.2	138.2	56.6	27.4
36) Toluene-d8 S2	surrogate	surrogate	surrogate	99.0	3.3	105.6	92.5	108.8	89.2	2.36
37) Toluene	5.2E+05	10.0	4.57	100.6	9.8	120.2	81.1	129.9	71.3	26.2
38) trans-1,3-Dichloropropene	7.8E+02	10.0	4.61	96.7	9.5	115.6	77.7	125.1	68.2	22.7
39) 1,1,2-Trichloroethane	7.3E+02	10.0	4.14	97.8	9.5	116.9	78.7	126.4	69.2	23.2
40) Tetrachloroethene	4.8E+02	10.0	8.41	98.1	12.7	123.4	72.7	136.1	60.0	31.0
41) 1.3-Dichloropropane	1.0E+05	10.0	4.21	99.3	9.0	117.4	81.3	126.4	72.3	22.5
42) 2-Hexanone	NA	100.0	4.46	97.1	15.6	128.3	65.9	143.9	50.3	28.4
43) Dibromochloromethane	1.1E+03	10.0	3.40	94.5	9.8	114.1	75.0	123.8	65.2	22.1
44) 1,2-Dibromoethane	3.2E+01	20.0	2.95	97.5	8.7	114.9	80.1	123.6	71.4	23.6
46) Chlorobenzene	1.5E+05	10.0	2.96	100.8	8.0	116.7	84.9	124.6	76.9	19.2
47) Ethylbenzene	4.0E+05	10.0	5.43	101.1	9.6	120.3	82.0	129.8	72.4	23.2
48) 1,1,1,2-Tetrachloroethane	3.2E+03	10.0	3.15	98.8	8.3	115.3	82.2	123.6	74.0	19.1
49) m & p - Xylene	2.7E+05	20.0	2.67	101.4	9.6	120.6	82.2	130.2	72.6	48.6
50) o-Xylene	2.7E+05	20.0	2.54	100.7	8.5	117.8	83.6	126.4	75.1	22.0
51) Styrene	1.7E+06	10.0	1.87	101.6	7.9	117.4	85.8	125.3	77.9	20.6
52) Bromoform	6.2E+04	10.0	3.91	93.5	11.2	115.9	71.1	127.0	59.9	24.3
53) Isopropylbenzene	NA	10.0	3.32	100.8	10.6	122.0	79.5	132.6	68.9	27.3

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54) 4-Bromofluorobenzene S3	surrogate	surrogate	surrogate	98.5	5.5	109.4	87.5	114.9	82.0	3.73
55) 1,1,2,2-Tetrachloroethane	4.1E+02	20.0	4.89	100.5	11.4	123.4	77.6	134.9	66.2	23.4
56) Bromobenzene	2.8E+04	10.0	2.48	100.7	7.8	116.3	85.1	124.2	77.3	19.7
57) n-Propylbenzene	2.4E+05	10.0	2.78	101.1	10.8	122.7	79.4	133.6	68.6	27.9
58) 1,2,3-Trichloropropane	3.4E+01	20.0	2.46	100.5	10.5	121.6	79.4	132.1	68.9	23.1
59) 2-Chlorotoluene	NA	10.0	2.69	100.6	9.2	118.9	82.2	128.1	73.1	24.0
60) 1,3,5-Trimethylbenzene	2.1E+04	10.0	2.68	100.8	10.1	121.0	80.6	131.1	70.5	26.5
61) 4-Chlorotoluene	NA	10.0	2.08	100.3	9.2	118.7	81.8	128.0	72.5	23.6
62) tert-Butylbenzene	3.9E+05	10.0	2.91	100.6	11.5	123.5	77.7	135.0	66.2	29.4
63) 1,2,4-Trimethylbenzene	5.2E+04	10.0	3.49	100.4	9.2	118.8	82.0	128.0	72.8	24.9
64) sec-Butylbenzene	2.2E+05	10.0	2.48	102.0	11.8	125.6	78.4	137.5	66.5	30.3
65) 4-Isopropyltoluene	NA	10.0	3.65	100.9	11.9	124.7	77.0	136.6	65.1	29.9
66) 1,3-Dichlorobenzene	5.3E+05	10.0	3.39	101.3	8.6	118.5	84.1	127.1	75.5	22.3
68) 1,4-Dichlorobenzene	3.4E+03	10.0	6.54	101.5	8.3	118.1	84.8	126.4	76.5	21.2
69) n-Butylbenzene	2.4E+05	10.0	3.74	102.4	12.6	127.6	77.1	140.2	64.5	32.2
70) 1,2-Dichlorobenzene	6.0E+05	10.0	3.69	102.0	7.7	117.3	86.6	125.0	78.9	21.1
71) 1,2-Dibromo-3-chloropropane	4.6E+02	20.0	9.20	96.6	13.1	122.8	70.4	135.9	57.2	27.8
72) 1,2,4-Trichlorobenzene	6.2E+04	20.0	3.16	99.2	10.0	119.2	79.3	129.2	69.3	25.1
73) Hexachlorobutadiene	6.2E+03	20.0	0.510	99.4	14.8	129.0	69.8	143.7	55.1	35.4
74) Naphthalene	NA	20.0	0.867	100.2	13.0	126.1	74.2	139.0	61.3	31.0
75) 1,2,3-Trichlorobenzene	NA	20.0	0.694	100.4	10.3	121.0	79.8	131.3	69.5	25.4

** indicates values based on laboratory prepared samples (proper soil to methanol ratio) with a 100% solid value.

Endyne, Inc.
160 James Brown Drive
Williston Vermont

Control Limits 2006 EPA 8270C - Ind. Standard as Percent Recovery - Solid Samples								
Parameter	Method Detection Limit ug/L	Reporting Limit ug/L	MEAN	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
Pyridine	50.94	400.0	64.1	17.2	98.5	29.8	115.7	12.6
N-Nitrosodimethylamine	25.15	400.0	77.9	15.2	108.4	47.4	123.7	32.2
2-Fluorophenol (E.S.)		0.0	72.9	17.8	108.6	37.2	126.5	19.4
Aniline	21.84	400.0	79.9	18.5	116.8	43.0	135.3	24.5
Phenol	20.77	200.0	96.1	15.5	127.1	65.1	142.6	49.5
2-Chlorophenol	11.20	200.0	93.2	12.2	117.6	68.8	129.9	56.5
Phenol-d5 (E.S.)		0.0	86.9	17.9	122.6	51.2	140.5	33.3
bis(2-chloroethyl)Ether	22.75	200.0	89.5	16.3	122.1	56.9	138.5	40.5
1,3-Dichlorobenzene	11.92	80.0	76.6	18.5	113.7	39.6	132.2	21.0
1,4-Dichlorobenzene	7.88	80.0	75.3	16.8	108.9	41.7	125.6	25.0
1,2-Dichlorobenzene	14.44	80.0	77.6	16.9	111.5	43.7	128.4	26.8
Benzyl Alcohol	19.90	800.0	92.5	24.7	141.9	43.1	166.6	18.4
bis(2-chloroisopropyl	18.29	200.0	89.5	14.3	118.0	60.9	132.2	46.7
2-Methylphenol	13.66	200.0	101.4	12.2	125.8	76.9	138.0	64.7
Hexachloroethane	15.04	200.0	72.6	17.3	107.2	38.1	124.5	20.8
N-Nitrosodi-n-propylamine	20.78	400.0	100.3	18.0	136.4	64.3	154.5	46.2
3&4- Methylphenol	28.72	200.0	95.1	14.3	123.7	66.5	138.0	52.2
Nitrobenzene-d5 (E.S.)		0.0	84.1	21.8	127.8	40.4	149.6	18.5
Nitrobenzene	23.01	200.0	82.4	12.6	107.6	57.2	120.2	44.6
N-nitrosopiperidine	18.53	400.0	99.4	12.8	125.0	73.7	137.9	60.9
Isophorone	27.65	80.0	92.6	24.1	140.7	44.5	164.8	20.5
2-Nitrophenol	19.67	400.0	93.8	16.2	126.2	61.3	142.4	45.1
2,4-Dimethylphenol	31.27	200.0	97.3	16.4	130.1	64.5	146.5	48.1
bis(2-chloroethoxy)Methane	21.52	200.0	92.2	15.7	123.6	60.9	139.3	45.2
2,4-Dichlorophenol	18.44	200.0	92.8	19.1	130.9	54.7	149.9	35.6
1,2,4-Trichlorobenzene	7.15	80.0	79.6	17.2	113.9	45.2	131.0	28.1
Naphthalene	**9.77**	**80.0**	**85.9**	**8.9**	**103.6**	**68.2**	**112.5**	**59.4**
4-Chloroaniline	15.89	200.0	85.8	9.8	105.4	66.2	115.3	56.3
2,6-Dichlorophenol	18.99	200.0	99.4	13.1	125.5	73.2	138.6	60.1
Hexachlorobutadiene	13.41	200.0	82.0	19.9	121.7	42.3	141.6	22.5
N-Nitrosodi-n-butylamine	22.42	200.0	110.7	15.7	142.2	79.3	157.9	63.6
2-Methylnaphthalene	**15.37**	**80.0**	**89.7**	**20.0**	**129.7**	**49.7**	**149.8**	**29.6**
4-Chloro-3-methylphen	29.70	400.0	103.1	20.9	144.9	61.4	165.7	40.5
1-Methylnaphthalene	**16.74**	**80.0**	**98.5**	**14.4**	**127.3**	**69.7**	**141.7**	**55.4**
Hexachlorocyclopentad	16.72	800.0	75.1	17.9	111.0	39.2	128.9	21.2
2,4,6-Trichlorophenol	16.86	400.0	89.2	18.4	125.9	52.5	144.3	34.1
2,4,5-Trichlorophenol	28.47	400.0	85.6	18.1	121.7	49.4	139.8	31.4
2-Fluorbiphenyl (E.S.)		0.0	87.7	13.7	115.0	60.4	128.6	46.7

Endyne, Inc.
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Control Limits 2006 EPA 8270C - Ind. Standard as Percent Recovery - Solid Samples								
Parameter	Method Detection Limit ug/L	Reporting Limit ug/L	MEAN	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
2-Chloronaphthalene	15.63	80.0	76.3	12.1	100.4	52.1	112.4	40.1
1-Chloronaphthalene	16.26	80.0	89.5	11.9	113.2	65.7	125.1	53.8
2-Nitroaniline	19.78	800.0	79.1	8.7	96.5	61.7	105.1	53.1
Acenaphthylene	**7.31**	**80.0**	**86.7**	**8.9**	**104.5**	**68.9**	**113.4**	**60.0**
Dimethylphthalate	12.58	200.0	96.2	14.6	125.3	67.1	139.9	52.5
2,6-Dinitrotoluene	18.83	200.0	88.4	17.5	123.5	53.3	141.0	35.7
Acenaphthene	**9.01**	**80.0**	**87.9**	**9.2**	**106.4**	**69.4**	**115.7**	**60.2**
3-Nitroaniline	17.01	800.0	88.4	14.2	116.9	60.0	131.1	45.7
2,4-Dinitrophenol	31.73	800.0	83.9	20.2	124.3	43.5	144.5	23.3
Dibenzofuran	9.93	80.0	83.9	13.3	110.5	57.3	123.8	44.0
2,4-Dinitrotoluene	26.33	200.0	100.9	25.3	151.5	50.2	176.9	24.8
1-Naphthylamine	5.34	400.0	89.8	24.0	137.7	41.9	161.6	17.9
2-Naphthylamine	12.21	400.0	86.8	24.3	135.4	38.3	159.7	14.0
Fluorene	**11.77**	**80.0**	**94.7**	**8.2**	**111.0**	**78.3**	**119.1**	**70.2**
4-Nitrophenol	36.04	400.0	93.0	24.3	141.5	44.4	165.7	20.2
Diethylphthalate	9.84	200.0	89.6	11.3	112.1	67.0	123.4	55.7
4-Chlorophenylphenylether	15.96	80.0	76.9	13.2	103.2	50.5	116.4	37.3
4-Nitroaniline	14.72	800.0	85.5	15.8	117.0	54.0	132.8	38.2
4,6-Dinitro-2-methylphenol	29.54	2000.0	101.8	22.5	146.8	56.8	169.3	34.3
Azobenzene	16.18	200.0	79.6	12.1	103.9	55.4	116.0	43.2
N-Nitrosodiphenylamine	8.34	200.0	90.8	10.3	111.4	70.3	121.7	60.0
2,4,6-Tribromophenol (E.S.)		0.0	91.4	18.3	128.1	54.7	146.4	36.4
4-Bromophenylphenylether	13.57	80.0	80.3	14.1	108.6	52.0	122.7	37.9
Hexachlorobenzene	12.71	200.0	78.8	14.3	107.5	50.2	121.8	35.9
Pentachlorophenol	20.00	800.0	76.0	17.2	110.4	41.6	127.7	24.3
Phenanthrene	**9.50**	**80.0**	**92.3**	**8.3**	**109.0**	**75.6**	**117.3**	**67.2**
Anthracene	**21.58**	**80.0**	**93.9**	**9.4**	**112.6**	**75.1**	**122.0**	**65.7**
Carbazole	64.83	400.0	91.0	13.8	118.5	63.4	132.3	49.6
Di-n-butylphthalate	27.42	200.0	104.7	11.0	126.8	82.6	137.8	71.6
Benzidine	6.54	400.0	41.4	10.7	62.9	20.0	73.6	9.3
Fluoranthene	**19.82**	**80.0**	**106.9**	**25.1**	**157.0**	**56.8**	**182.0**	**31.7**
Pyrene	**16.01**	**80.0**	**106.6**	**24.3**	**155.1**	**58.1**	**179.4**	**33.8**
Terphenyl-d14 (E.S.)		0.0	112.5	14.8	142.1	82.8	157.0	68.0
Butylbenzylphthalate	16.02	200.0	110.6	14.2	138.9	82.2	153.1	68.0
Benzo(a)anthracene	**13.51**	**80.0**	**97.7**	**9.9**	**117.4**	**78.0**	**127.3**	**68.1**
Chrysene	**18.37**	**80.0**	**100.9**	**9.4**	**119.6**	**82.1**	**129.0**	**72.7**
3,3'-Dichlorobenzidin	17.01	200.0	93.3	18.0	129.4	57.3	147.4	39.3
bis(2-ethylhexyl)phth	15.32	200.0	107.6	27.3	162.2	53.1	189.5	25.8

Endyne, Inc.
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Control Limits 2006 EPA 8270C - Ind. Standard as Percent Recovery - Solid Samples								
Parameter	Method Detection Limit ug/L	Reporting Limit ug/L	MEAN	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
Di-n-octylphthalate	12.64	200.0	91.6	19.5	130.7	52.6	150.2	33.0
Benzo(b&k)fluoranthrenes	**11.96**	**80.0**	**93.5**	**8.7**	**110.9**	**76.1**	**119.6**	**67.4**
Benzo(a)pyrene	**19.82**	**80.0**	**92.3**	**8.6**	**109.5**	**75.1**	**118.1**	**66.5**
Indeno[1,2,3-cd]pyrene	**14.75**	**80.0**	**91.9**	**11.3**	**114.6**	**69.3**	**125.9**	**58.0**
Dibenz(a,h)anthracene	**17.37**	**80.0**	**91.0**	**11.8**	**114.5**	**67.4**	**126.2**	**55.7**
Benzo(g,h,i)perylene	**25.01**	**80.0**	**87.7**	**12.1**	**112.0**	**63.4**	**124.2**	**51.3**

Bolded parameters indicate PAH test group

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Endyne, Inc.
160 James Brown Drive
Williston, Vermont

Control Limits 2007 EPA 8081A - Ind. Standard as Percent Recovery - Solid Samples

Parameter	Method Detection Limit ug/kg	Reporting Limit ug/kg	MEAN ug/kg	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
BHC, alpha-	2.04	< 10.0	10.69	2.77	20.54	9.44	23.31	6.66
BHC, gamma-	1.76	< 10.0	11.05	2.75	20.98	9.97	23.73	7.21
BHC, beta-	1.88	< 10.0	12.50	3.17	23.61	10.92	26.78	7.75
BHC, delta-	2.55	< 10.0	12.86	2.63	21.96	11.44	24.59	8.81
Heptachlor	2.90	< 10.0	11.35	3.67	23.88	9.21	27.54	5.54
Aldrin	2.18	< 10.0	10.38	2.57	19.78	9.49	22.35	6.92
Hept Epoxide	2.42	< 10.0	16.58	2.71	22.00	11.15	24.72	8.44
Endosulfan I	2.64	< 10.0	16.65	2.57	21.80	11.50	24.37	8.93
DDE	2.26	< 10.0	17.07	2.67	22.41	11.72	25.09	9.05
Dieldrin	2.51	< 10.0	17.81	3.27	24.34	11.27	27.61	8.00
Endrin	2.40	< 10.0	17.71	3.06	23.82	11.59	26.87	8.54
Endosulfan II	2.78	< 10.0	17.16	3.42	24.00	10.31	27.42	6.89
DDD	5.41	< 10.0	17.90	3.07	24.05	11.76	27.12	8.69
Endrin Aldehyde	2.81	< 10.0	16.23	2.85	21.93	10.53	24.78	7.68
Endo Sulfate	4.27	< 10.0	17.03	3.63	24.30	9.77	27.93	6.13
DDT	3.51	< 10.0	18.08	3.27	24.63	11.54	27.90	8.26
Methoxychlor	5.39	< 20.0	18.61	2.97	24.55	12.66	27.52	9.69
Chlordane	2.34	< 20.0	66.60				86.6	46.6
Toxaphene	2.36	< 50.0	330				231	429
DCB (Surrogate)	NA	NA	30.60	4.09	38.783	22.417	42.874	18.326

Endyne, Inc.
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Control Limits 2007 EPA 8081A - Ind. Standard as Percent Recovery - Solid Samples

Parameter	Method Detection Limit ug/kg	Reporting Limit ug/kg	MEAN ug/kg	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
BHC, alpha-	2.04	< 10.0	10.69	2.77	20.54	9.44	23.31	6.66
BHC, gamma-	1.76	< 10.0	11.05	2.75	20.98	9.97	23.73	7.21
BHC, beta-	1.88	< 10.0	12.50	3.17	23.61	10.92	26.78	7.75
BHC, delta-	2.55	< 10.0	12.86	2.63	21.96	11.44	24.59	8.81
Heptachlor	2.90	< 10.0	11.35	3.67	23.88	9.21	27.54	5.54
Aldrin	2.18	< 10.0	10.38	2.57	19.78	9.49	22.35	6.92
Hept Epoxide	2.42	< 10.0	16.58	2.71	22.00	11.15	24.72	8.44
Endosulfan I	2.64	< 10.0	16.65	2.57	21.80	11.50	24.37	8.93
DDE	2.26	< 10.0	17.07	2.67	22.41	11.72	25.09	9.05
Dieldrin	2.51	< 10.0	17.81	3.27	24.34	11.27	27.61	8.00
Endrin	2.40	< 10.0	17.71	3.06	23.82	11.59	26.87	8.54
Endosulfan II	2.78	< 10.0	17.16	3.42	24.00	10.31	27.42	6.89
DDD	5.41	< 10.0	17.90	3.07	24.05	11.76	27.12	8.69
Endrin Aldehyde	2.81	< 10.0	16.23	2.85	21.93	10.53	24.78	7.68
Endo Sulfate	4.27	< 10.0	17.03	3.63	24.30	9.77	27.93	6.13
DDT	3.51	< 10.0	18.08	3.27	24.63	11.54	27.90	8.26
Methoxychlor	5.39	< 20.0	18.61	2.97	24.55	12.66	27.52	9.69
Chlordane	2.34	< 20.0	66.60				86.6	46.6
Toxaphene	2.36	< 50.0	330				231	429
DCB (Surrogate)	NA	NA	30.60	4.09	38.783	22.417	42.874	18.326

Endyne, Inc.
160 James Brown Drive
Williston, Vermont

Control Limits 2007 EPA 8081A - Ind. Standard as Percent Recovery - Solid Samples

Parameter	Method Detection Limit ug/kg	Reporting Limit ug/kg	MEAN ug/kg	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
BHC, alpha-	2.04	< 10.0	10.69	2.77	20.54	9.44	23.31	6.66
BHC, gamma-	1.76	< 10.0	11.05	2.75	20.98	9.97	23.73	7.21
BHC, beta-	1.88	< 10.0	12.50	3.17	23.61	10.92	26.78	7.75
BHC, delta-	2.55	< 10.0	12.86	2.63	21.96	11.44	24.59	8.81
Heptachlor	2.90	< 10.0	11.35	3.67	23.88	9.21	27.54	5.54
Aldrin	2.18	< 10.0	10.38	2.57	19.78	9.49	22.35	6.92
Hept Epoxide	2.42	< 10.0	16.58	2.71	22.00	11.15	24.72	8.44
Endosulfan I	2.64	< 10.0	16.65	2.57	21.80	11.50	24.37	8.93
DDE	2.26	< 10.0	17.07	2.67	22.41	11.72	25.09	9.05
Dieldrin	2.51	< 10.0	17.81	3.27	24.34	11.27	27.61	8.00
Endrin	2.40	< 10.0	17.71	3.06	23.82	11.59	26.87	8.54
Endosulfan II	2.78	< 10.0	17.16	3.42	24.00	10.31	27.42	6.89
DDD	5.41	< 10.0	17.90	3.07	24.05	11.76	27.12	8.69
Endrin Aldehyde	2.81	< 10.0	16.23	2.85	21.93	10.53	24.78	7.68
Endo Sulfate	4.27	< 10.0	17.03	3.63	24.30	9.77	27.93	6.13
DDT	3.51	< 10.0	18.08	3.27	24.63	11.54	27.90	8.26
Methoxychlor	5.39	< 20.0	18.61	2.97	24.55	12.66	27.52	9.69
Chlordane	2.34	< 20.0	66.60				86.6	46.6
Toxaphene	2.36	< 50.0	330				231	429
DCB (Surrogate)	NA	NA	30.60	4.09	38.783	22.417	42.874	18.326

Endyne, Inc.
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Control Limits 2007 EPA 8082 - Ind. Standard as Percent Recovery - Aqueous Samples

Parameter	Method Detection Limit ug/L	Reporting Limit ug/L	MEAN	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
Arochlor-1016	0.152	< 0.50	2.94	0.56	4.06	1.82	4.63	1.26
Arochlor-1221		< 0.50	3.00				3.60	2.40
Arochlor-1232		< 0.50	3.00				3.60	2.40
Arochlor-1242		< 0.50	3.00				3.60	2.40
Arochlor-1248		< 0.50	3.00				3.60	2.40
Arochlor-1254		< 0.50	3.00				3.60	2.40
Arochlor-1260	0.111	< 0.50	3.03	0.41	3.99	2.06	4.47	1.58
Arochlor-1268		< 0.50	3.00				3.60	2.40
DCB (Surrogate)	NA	NA	0.950	0.170	1.286	0.604	1.456	0.434

Control Limits 2007 EPA 8082 - Ind. Standard as Percent Recovery - Solid Samples

Parameter	Method Detection Limit ug/kg	Reporting Limit ug/kg	MEAN ug/kg	Standard Deviation	Upper Warning Limit	Lower Warning Limit	Upper Control Limit	Lower Control Limit
Arochlor-1016	3.37	< 50.0	2.89	0.55	3.99	1.78	4.54	1.23
Arochlor-1221		< 50.0	100				120	80
Arochlor-1232		< 50.0	100				120	80
Arochlor-1242		< 50.0	100				120	80
Arochlor-1248		< 50.0	100				120	80
Arochlor-1254		< 50.0	100				120	80
Arochlor-1260	1.96	< 50.0	2.82	0.36	3.54	2.09	3.91	1.72
Arochlor-1268		< 50.0	100				120	80
DCB (Surrogate)	NA	NA	0.93	0.27	1.469	0.381	1.741	0.109

Endyne, Inc.
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2006 Quality Control for PRIORITY POLLUTANT 13 METALS in Water

Element	Method	MDL ug/L	Reporting Limit ppb	Independent LFB Accuracy Limit %	Precision Limit %
Antimony	SM 3113B	0.82	2	10	20
Arsenic	SM 3113B	0.57	2	10	20
Beryllium	EPA 6010	0.3	2	10	20
Cadmium	EPA 6010	0.4	3	10	20
Chromium	EPA 6010	4.4	10	10	20
Copper	EPA 6010	2.43	10	10	20
Lead	SM 3113B	0.26	2	10	20
Mercury	EPA 7470	0.22	1	10	20
Nickel	EPA 6010	5.17	20	10	20
Selenium	SM 3113B	1.73	5	10	20
Silver	EPA 6010	4.53	10	10	20
Thallium	SM 3113B	0.36	1	10	20
Zinc	EPA 6010	2.16	20	10	20

2006 Quality Control for PRIORITY POLLUTANT 13 METALS in Soil

Element	Method	MDL mg/Kg	Reporting Limit mg/kg	Independent LFB Accuracy Limit %	Duplicate Precision Limit %
Antimony	EPA 6010	1.09	2.00	10.00	20.00
Arsenic	EPA 6010	0.676	2.00	10.00	20.00
Beryllium	EPA 6010	0.144	0.50	10.00	20.00
Cadmium	EPA 6010	0.150	0.50	10.00	20.00
Chromium	EPA 6010	0.257	1.00	10.00	20.00
Copper	EPA 6010	0.235	1.00	10.00	20.00
Lead	EPA 6010	0.468	2.00	10.00	20.00
Mercury	EPA 7471	0.300	0.600	10.00	20.00
Nickel	EPA 6010	0.334	1.50	10.00	20.00
Selenium	EPA 6010	1.01	3.00	10.00	20.00
Silver	EPA 6010	0.373	2.00	10.00	20.00
Thallium	EPA 6010	0.938	1.00	10.00	20.00
Zinc	EPA 6010	0.373	1.50	10.00	20.00

APPENDIX I

Dissolved Oxygen Concentration Measurements & Water Sampling/Phosphorus Analysis Standard Operating Procedures (SOP)

By Jennifer Drociak

A. Dissolved Oxygen Instrument/Equipment Testing, Inspection, Maintenance, Calibration and Frequency.

Dissolved oxygen will be monitored once a month during the months of June, July, and August.

Dissolved oxygen will be measured utilizing a YSI Model 85 hand-held multimeter. Protocols are as follows:

Checking the Membrane:

To ensure the probe remains moist inside the meter calibration/storage chamber, pull the probe out of the chamber and add a few drops of deionized (DI) water to the sponge at the bottom of the calibration/storage chamber. Turn the meter on its side to allow any excess water to drain out of the chamber. *This step will only be necessary once per sampling day, but be sure the sponge in the calibration/storage chamber is moist before storage.* Be careful not to over-wet the sponge. *Calibrate the meter after 15 minutes and after any excess "puddled" water is drained from the chamber.*

This step should be completed before the meter is turned on.

Note: The wet sponge creates a 100% saturated air environment within the chamber for ideal calibration conditions. Ensure that the sensor does not contact the wet sponge by inserting the probe only until the rubber seal is flush with the outer edge of the chamber.

If Bubbles ARE Detected

If bubbles are detected or if membrane is due for routine replacement, follow Step a through Step g below to replace the membrane. (Record the performed maintenance under the "Comments" section on the back of the VRAP Field Data Sheet.) **Otherwise proceed with Step 1 below.**

a. Unscrew and remove the black protective cage.

b. Unscrew and remove the old membrane cap.

c. Thoroughly rinse the sensor tip (gold and silver areas) with DI water.

d. Hold the membrane cap upside down and add enough drops of the membrane probe solution to form a meniscus in the probe membrane cap.

e. Tap the bottom of the cap with your finger a few times to remove any trapped air bubbles. **CAUTION:** Do not touch the membrane surface.

f. Screw the membrane cap onto the probe tightly by hand (to prevent leakage of probe solution). A small amount of probe solution should overflow.

g. Shake off any excess probe solution and rinse the sensor thoroughly with distilled water to prevent corrosion.

If Bubbles Are Not Detected

Calibration:

1. Turn the meter on by pressing the **ON/OFF** button. **Ensure the meter has been turned ON for at least 15 minutes before calibrating.** Record the time the dissolved oxygen meter was turned on - on the upper right front page of the VRAP Field Data Sheet. If 15 minutes have not yet elapsed, you may move on to calibration procedures for the other equipment.

2. Record the time of the first dissolved oxygen calibration on the upper right front page of the VRAP Field Data Sheet.

3. Press the **MODE** button until dissolved oxygen is displayed in % saturation. The **UP** button on the YSI 95 and the **MODE** button and YSI 85 can be used to toggle back and forth between % saturation and mg/L, however, the YSI 85 will also cycle through the specific conductance modes.

4. Press and release both the **DOWN** and **UP** arrow buttons (DOWN slightly prior to UP) to enter the DO/Temperature meter calibration menu. You will see **CAL** in the lower left hand corner when you have successfully entered calibration mode.

5. The screen will prompt you to enter the local altitude in hundreds of feet. Use the **UP** and **DOWN** arrows to adjust the reading appropriately (For example, entering a 12 indicates 1200 feet above sea level) and press **ENTER**.

6. If using the YSI 95, the screen will prompt you to enter the salinity of the sample you will be measuring. Be sure the screen reads zero and press **ENTER**.

7. **Record** the calibration value (displayed on the bottom right-corner of the LCD screen) on the VRAP Field Data Sheet. The calibration value will vary with altitude and thus may be different at each station if the altitude varies.

8. Press **ENTER** again. The display should read **SAVE** and then return to normal measurement mode. This value will now be the calibration value.

9. Watch to ensure that this value does not drift (i.e. goes up or down more than 3%). If drift occurs first check the sponge in the chamber to ensure that it is saturated.

10. **Leave the meter on until you are finished with all measurements for the day. Calibration must be repeated before <u>each</u> individual measurement. If the meter shuts off, you must wait 15 minutes with the probe in its chamber before calibrating.**

Dissolved Oxygen Probe Precautions

- Membrane life depends on usage. Membranes will last a long time if installed properly and treated with care. Erratic readings are a result of loose, wrinkled, damaged, or fouled membranes, or from large bubbles in the electrolyte reservoir. If erratic readings or evidence of membrane damage occurs, you should replace the membrane and the solution.

- To keep the membrane from drying out, store the probe in the calibration chamber with the damp sponge.

Sampling

Note: The Dissolved Oxygen/Temperature meter must be calibrated prior to each DO measurement. Both the YSI 95 and YSI 85 hand-held multimeters are calibrated at every station (for % saturation) prior to sampling during the day to the altitude in nearest 100 feet. The meters are checked only once a day (not calibrated) against the Zero Oxygen Dissolved Oxygen standard in both percent saturation and concentration in mg/L. Below are the specifications of the meter that will be used in the field under the Volunteer River Assessment Program (VLAP), which is a YSI 85 hand-held multimeter. The meter reads -5 to 65 degrees Celsius for temperature and 0-20 mg/L/0-200% for dissolved oxygen.

The process for checking the Zero Dissolved Oxygen Standard would be done once a day (at any time during the day), where the probe is placed in a 250 mL bottle of standard (Standard Methods 4500-O G : Sodium sulfite + Deionized water), and the dissolved oxygen % saturation and concentration in mg/L is recorded after the value has stabilized on the LCD screen (typically around 2 minutes). The standard should read at or below 0.5 mg/L. This is done as one of the "QA/QC Meter Checks". Thus, the meters get calibrated at the high end of the range, and checked at the low end of the range.

Dissolved Oxygen (% Saturation and mg/L)

1. Remove the probe from the calibration chamber and rinse the probe and cable (approximately 6 inches) with DI water. If necessary, press the **MODE** button until dissolved oxygen is displayed in % saturation.
2. Submerge the probe about 2/3 of the way into the bucket and agitate by slowly moving the probe back and forth in the sample for a minimum of two minutes for the temperature and dissolved oxygen readings to stabilize. Some bouncing will occur due to the probes' sensitivity. Record the water temperature (°C) and the DO (% saturation then mg/L) on the VRAP Field Data Sheet. (Press the **UP** button on the YSI 95 or the **MODE** button on the YSI 85 to toggle between % saturation to mg/L.)
3. To determine the air temperature (°C), remove the probe from the sample and hold in the air until the temperature reading to stabilizes**. It should only take a minute for the air temperature reading to stabilize. Do not leave the probe out of the chamber longer as it will damage the membrane.**
4. Rinse the probe with DI water and return it to the storage chamber.
5. Press **UP** button on the YSI 95 (or **MODE** button on the YSI 85) to toggle back to % saturation.
6. Wait two minutes and **record** the dissolved oxygen % saturation on the VRAP Field Data Sheet in the column marked "Dissolved Oxygen % Saturation in Chamber".
7. The Dissolved Oxygen/Temperature meter should remain on until the last station has been sampled. If the meter is turned off prior to the end of the sampling day, the meter must be turned on and allowed a 15-minute warm-up period, with the probe in its chamber, prior to calibration and additional sampling.
8. Rinse the probe with DI water before returning it to the calibration chamber.

B. Phosphorous Instrument/Equipment Testing, Inspection, Maintenance, Calibration and Frequency.

Total phosphorus will be collected by Jen Drociak and will be analyzed by the NHDES Laboratory Services via EPA Method 365.3

Total phosphorus samples will be collected as follows:

VRAP

1. Carefully wade out into the river as close as possible to the center. Do not enter water above your waist, and do not enter the water if there is any concern for your safety. Be sure to have someone on shore that knows where you are. NHDES highly recommends that volunteers wear an appropriate personal floatation device when working in or near the water.
2. Position yourself facing upstream and rinse the bucket in the river three times. **Do not collect the water that is running over your legs/boots.** Discard the rinse water behind yourself.
3. With the bucket facing upstream and held in front of your body, slowly dip the lip of the bucket into the flowing water and allow the bucket to fill. Rivers receive oxygen from the atmosphere through mixing. Just as riffles and rapids increase the oxygen in a river or stream, rushing water over the side of the bucket will add oxygen to the sample and yield inaccurate readings.
4. Carefully return to shore with the bucket ½ - ¾ full and place it on the bank for immediate analysis. Do not place the bucket on the hot tar/asphalt or in direct sunlight.

Preserve the bottles properly (in a cooler on ice), and submit the samples to the NHDES Laboratory Services within the sample holding time appropriate to each test. Always return samples to the NHDES Laboratory Services before 3 pm (Monday – Thursday) or before 12 pm on Friday.

VLAP

- Do not sample if the tributary is not flowing or too shallow. Stagnant water will result in values which are not representative of the water entering or leaving the lake.
- Do not sample an area if the bottom sediments have recently been disturbed. You may sample upstream in an undisturbed area. Sediment particles in a sample will complicate and sometimes invalidate laboratory analysis.
- Check your old reports and lake map to repeat the same sampling stations each month.
- Label one (1) large white and one (1) small brown bottle per sampling station.
- Rinse the large white bottle by scooping into the flow upstream *(away from you),* then shake and discard the water downstream.
- Fill the large white bottle to the neck.
- Pour from the large white bottle and fill the small brown bottle to the neck. ***Do not rinse or overflow the small brown bottle. It contains sulfuric acid as a preservative!***
- Top off the large white bottle and place the sample in a cooler with ice.

- Repeat the steps with all tributary sampling stations.

Preserve the bottles properly (in a cooler on ice), and submit the samples to the NHDES Laboratory Services within the sample holding time appropriate to each test. Always return samples to the NHDES Laboratory Services before 3 pm (Monday – Thursday) or before 12 pm on Friday.

Please refer to the existing approved QAPPS for the Volunteer Lake Assessment Program (VLAP) and the Volunteer River Assessment Program (VRAP) for additional information.

Measurement Performance Criteria

Precision is calculated for field and laboratory measurements through measurement replicates (instrumental variability) and is calculated for each sampling day. The use of VRAPdata for assessment purposes is contingent on compliance with a parameter-specific relative percent difference (RPD) as derived from equation 1, below. Any data exceeding the limits of the individual measures are disqualified from surface water quality assessments. All data that exceeds the limits defined by the VRAPQAPPare acknowledged in the data tables with an explanation of why the data was unusable. Table 1 shows typical parameters studied under VRAPand the associated quality control procedures.

(Equation 1)

where x1 is the original sample andx2 is the replicate sample

Table 1. Field Analytical Quality Controls

Water Quality Parameter	QC Check	QC Acceptance Limit	Corrective Action	Person Responsible for Corrective Action	Data Quality Indicator
Temperature	Measurement Replicate	RPD < 10% or Absolute Difference <0.8 C.	Repeat Measurement	Volunteer Monitors	Precision
Dissolved Oxygen	Measurement Replicate	RPD < 10%	Recalibrate Instrument, Repeat Measurement	Volunteer Monitors	Precision
	Known Buffer (Zero O_2 Sol.)	RPD < 10% or Absolute Difference <0.4 mg/L	Recalibrate Instrument, Repeat Measurement	Volunteer Monitors	Relative Accuracy
pH	Measurement Replicate	RPD < 10% or Absolute Difference <0.3 pH units	Recalibrate Instrument, Repeat Measurement	Volunteer Monitors	Precision
	Known Buffer (pH = 6.0)	±0.1 std units	Recalibrate Instrument, Repeat Measurement	Volunteer Monitors	Accuracy
Specific Conductance	Measurement Replicate	RPD < 10% or Absolute Difference <5µS/cm	Recalibrate Instrument, Repeat Measurement	Volunteer Monitors	Precision
	Method Blank (Zero Air Reading)	±5.0 µS/cm	Recalibrate Instrument, Repeat Measurement	Volunteer Monitors	Accuracy
Turbidity	Measurement Replicate	RPD < 10% or Absolute Difference <0.5 NTU	Recalibrate Instrument, Repeat Measurement	Volunteer Monitors	Precision
	Method Blank (DI Water)	±0.1 NTU	Recalibrate Instrument, Repeat Measurement	Volunteer Monitors	Accuracy
Laboratory Parameters	Measurement Replicate	RPD < 20% or Absolute Difference less than ½ the mean value of the parameter in NHDES's Environmental Monitoring Database	Repeat Measurement	Volunteer Monitors	Precision