

91346

# DELINEATION SAMPLING PROGRAM



ASHTABULA PLANT II  
ASHTABULA, OHIO



APRIL 1997  
Revision 2

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1

Lancaster Laboratories, Inc.  
Standard Operating Procedure  
SW846 Method 8081

**EXECUTIVE  
SUMMARY**

## EXECUTIVE SUMMARY

Numerous soil samples for polychlorinated biphenyl (PCB) analysis have been collected at the Millennium Inorganic Chemicals Inc., Plant II  $TiCl_4$  facility (formerly the SCM Chemicals, Inc. Plant II  $TiCl_4$  facility). Samples have been collected from December 1990 to present under a Toxic Substances Control Act (TSCA) work plan, under the Source Control Operable Unit (SCOU) investigation, and as part of site construction activities. The PCB data were used to evaluate the lateral and vertical extent of PCBs within the facility for better definition of remedial alternatives in the Feasibility Study (FS).

A Delineation Sampling Program has been planned in order to further define the response areas that were developed in the SCOU FS for Alternative VI and further delineate the extent of PCBs to support the design process. This will ensure that the response areas are accurately depicted and also help to prevent unnecessary remedial activities in clean areas.

The sampling strategy will be based on a systematic approach that includes additional soil sampling on a 50-foot grid in areas that drain directly to Fields Brook and on a 100-foot grid in active plant areas that drain to the facility stormwater collection area. This sampling program will supplement the systematic sampling already performed at the facility.

The Millennium facility presently has over 150 borings representing 750 PCB analyses. The sampling approach defined in the following sections provides for approximately 43 additional borings and 19 additional surface samples. Thus, after implementation of this sampling program, over 1,000 PCB analyses will have been performed at the 28-acre Millennium facility. The proposed number of samples is considered adequate by United States Environmental Protection Agency (USEPA) for further delineation of PCBs in both non-response areas which have not been sampled and the remedial response areas which were identified in the SCOU FS, Alternative VI.





## SECTION 1

### ADDITIONAL DATA NEEDS

Numerous soil samples for polychlorinated biphenyl (PCB) analysis have been collected at the Millennium Inorganic Chemicals Inc. Plant II  $TiCl_4$  facility (formerly the SCM Chemicals, Inc. Plant II  $TiCl_4$  facility). Samples have been collected from December 1990 to present under a Toxic Substances Control Act (TSCA) work plan, under the Source Control Operable Unit (SCOU) investigation, and as part of site construction activities. The PCB data were used to evaluate the lateral and vertical extent of PCBs within the facility for better definition of remedial alternatives in the Feasibility Study (FS). Please refer to the February 20, 1997 letter to the United States Environmental Protection Agency (USEPA) from **AquaEter** for a full description of contamination and historical sampling events.

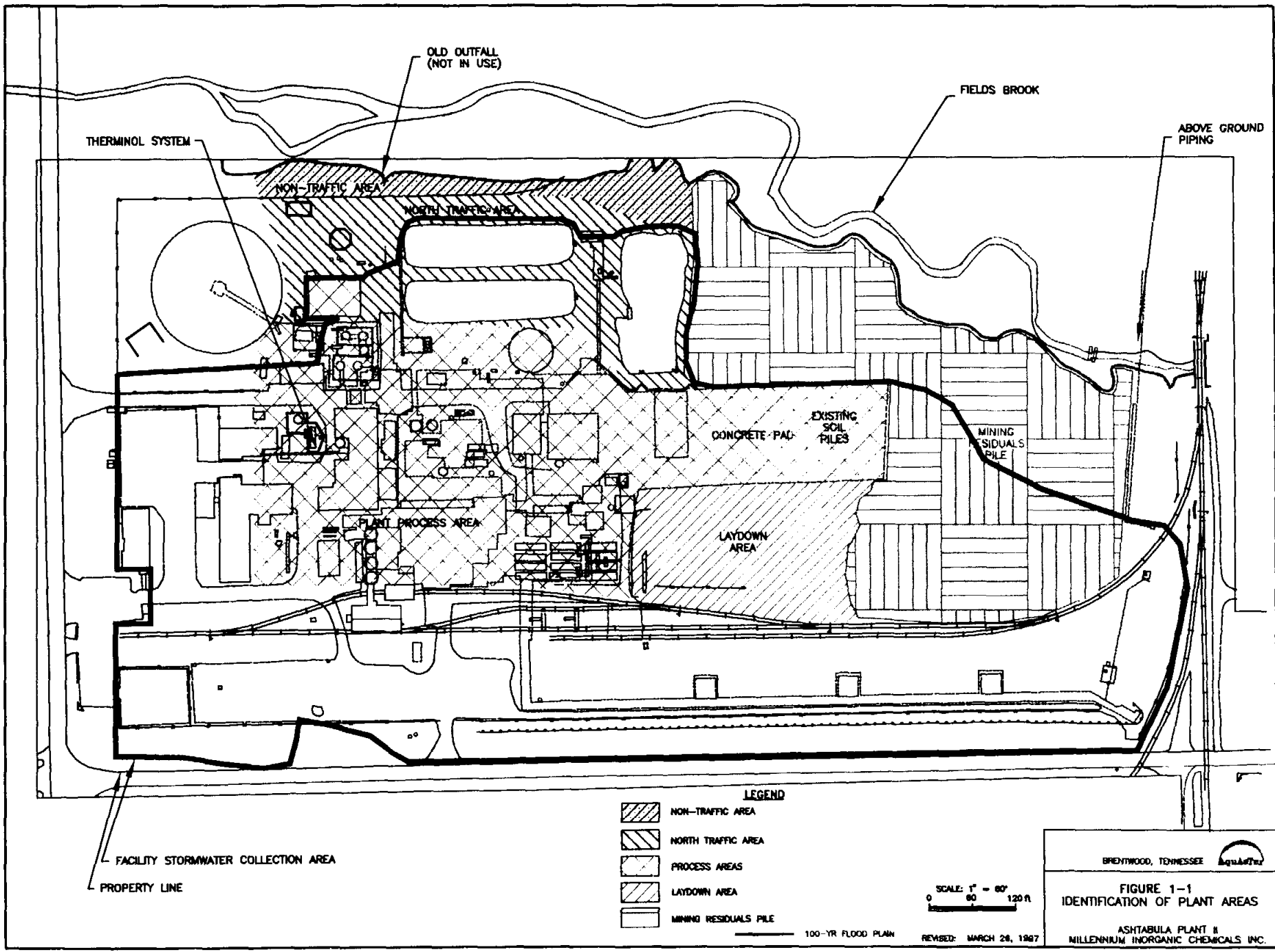
As described in the February 1997 SCOU FS, Alternative VI consists of remedial actions in five facility areas, as presented in Figure 1-1: **1) the Non-Traffic Area; 2) the North Traffic Area; 3) the Laydown Area; 4) the Plant Process Area; and 5) the Mining Residuals Pile.** Under Alternative VI, remediation would include the following:

- ◆ excavation of materials with greater than 50 mg/kg PCBs and replacement with clean soils (where necessary);
- ◆ placement of cover materials (asphalt, concrete, gravel, or 12 inches of soil and vegetation) over areas where PCBs have been detected between 3.1 and 50 mg/kg;
- ◆ disposal in an approved facility; and
- ◆ long-term maintenance of the site.

The response areas for Alternative VI are thus areas with greater than 50 mg/kg PCBs (for excavation and disposal) and areas with 3.1 to 50 mg/kg PCBs (for placement of cover). In order to further define the response areas that were developed in the SCOU FS for Alternative VI, USEPA has requested that Millennium further delineate the extent of PCBs to support the design process. This will ensure that the response areas are accurately depicted and also help to prevent unnecessary remedial activities in clean areas.

Therefore, a Delineation Sampling Program (DSP) has been planned and supported by USEPA. The sampling strategy will be based on a systematic approach that includes additional soil sampling on a 50-foot grid in areas that drain directly to Fields Brook and on a 100-foot grid in active plant areas that drain to the Facility Stormwater Collection Area (FSCA). This sampling program will supplement the systematic sampling already performed at the facility.

The Millennium facility presently has over 150 borings representing 750 PCB analyses. The sampling approach defined in the following sections provides for approximately 47 additional borings and 15 additional surface samples, for a total of 310 PCB analyses. Thus, after implementation of this sampling program, over 1,000 PCB analyses will have been performed at the 28-acre Millennium facility. The proposed number of samples is considered adequate by USEPA for further delineation of PCBs in both non-response areas which have not been sampled and the remedial response areas which were identified in the SCOU FS, Alternative VI.





## SECTION 2

### SAMPLE LOCATIONS

#### PLANT AREA IDENTIFICATION

Delineation sampling at the  $\text{TiCl}_4$  facility will address several different plant areas, both inside and outside of the FSCA, as previously presented in Figure 1-1. Facility areas associated with potential or known PCB use, transport, or disposal (i.e., the old Therminol system) are presented in Figure 2-1. Facility areas associated or potentially associated with the incidental vehicular movement of PCBs (i.e., roadways) are presented in Figure 2-2. Facility areas with no known or potential connection to PCB use, transport, or disposal are presented in Figure 2-3.

#### DELINEATION SAMPLING LOCATIONS

The following text describes the general basis of the delineation sampling, as presented in Table 2-1 and Figure 2-4.

Outside the FSCA, samples will be collected on a 50-foot grid, every two feet vertically to till (samples X2, X11 to X22 and X24 to X43). However, certain locations will consist only of surface samples (samples X1, X6, X7, and X23). Samples X14, X18, X27, X28, X31, and X32 are expected to be placed using the drill rig. Drilling locations will be marked and the individual locations along Fields Brook will be assessed as to whether they are accessible. If field observations indicate that the locations are too steep for safe use of the drill rig, a field decision will be made to convert these to hand-augured surface samples.

Inside the FSCA in the Mining Residuals Pile, samples will be collected on a 100-foot grid, every 4 feet vertically to the soil/till interface (samples Z15 and Z16).

Inside the FSCA in the Laydown Area, samples will be collected on a 100-foot grid, to a depth of 4 feet. The depth range for soil analysis from the core samples from these three borings has been randomly selected. Location Z9 will be analyzed from 0 to 2 feet, location Z6 will be analyzed from 1 to 3 feet, and location Z10 will be analyzed from 2 to 4 feet.

Two samples will be collected from inside the FSCA under the concrete pad (Z3 and Z4). The actual samples will be collected from a depth of 0 to 2 feet; however, the boring will be progressed to a depth of 7 feet, and a boring log will be kept to confirm that mining residuals are not encountered.

Inside the FSCA in the remainder of the plant, samples will be collected on a 100-foot grid. Surface samples will be collected in locations Z1, Z2, Z7, Z8, and Z11 to Z14). Location Z5 will be sampled every four feet vertically to the soil/till.

In addition, inside the FSCA, but in areas with no known or potential connection to PCB use, transport, or disposal (i.e., to the south of the railroad tracks and outside the fence in the west parking/grass areas), three random surface samples will be collected (R1, R2, and R3).

The proposed plan includes 47 locations for soil borings with a drill rig (to an average depth of 14 feet), 15 surface sampling locations, and a total of 310 PCB analyses (including quality assurance/quality control analyses). The sample locations shown in Figure 2-4 are approximate and may be relocated within their respective grid areas, if necessary, to allow sampling around structures and utilities.

TABLE 2-1. DELINEATION SAMPLING PROGRAM

BORING ID	BORING DEPTH (ft)	NUMBER OF SAMPLES
X1	surface	1
X2	14	7
X3	14	7
X4	14	7
X5	14	7
X6	surface	1
X7	surface	1
X8	14	7
X9	14	7
X10	14	7
X11	16	8
X12	18	9
X13	14	7
X14	16	8
X15	14	7
X16	14	7
X17	12	6
X18	14	7
X19	20	10
X20	18	9
X21	14	7
X22	12	6
X23	surface	1
X24	14	7
X25	14	7
X26	20	10
X27	20	10
X28	6	3
X29	18	9
X30	14	7
X31	16	8
X32	16	8
X33	16	8
X34	14	7
X35	6	3
X36	16	8
X37	14	7
X38	12	6
X39	8	4
X40	16	8
X41	10	5
X42	16	8
X43	14	7

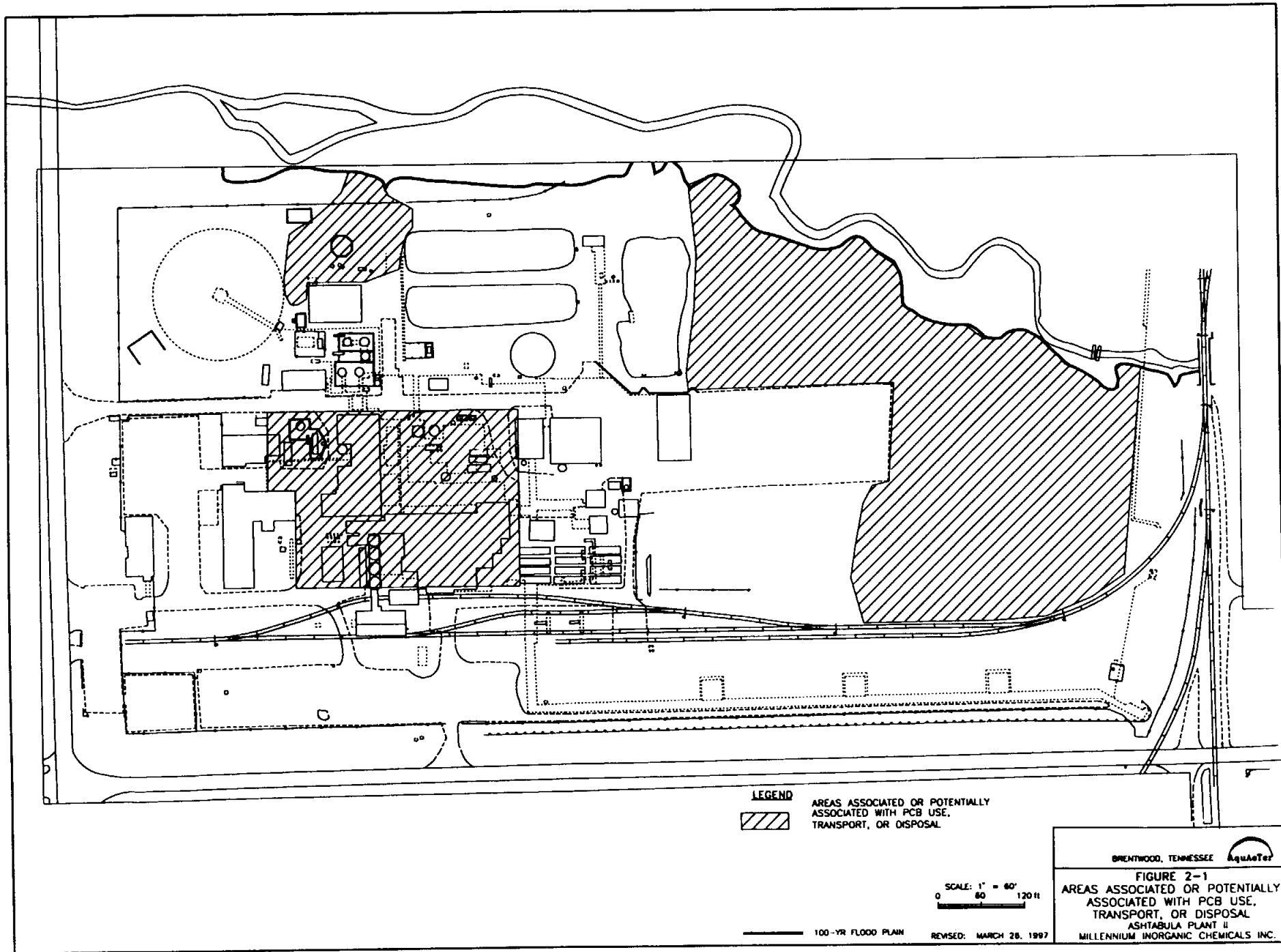


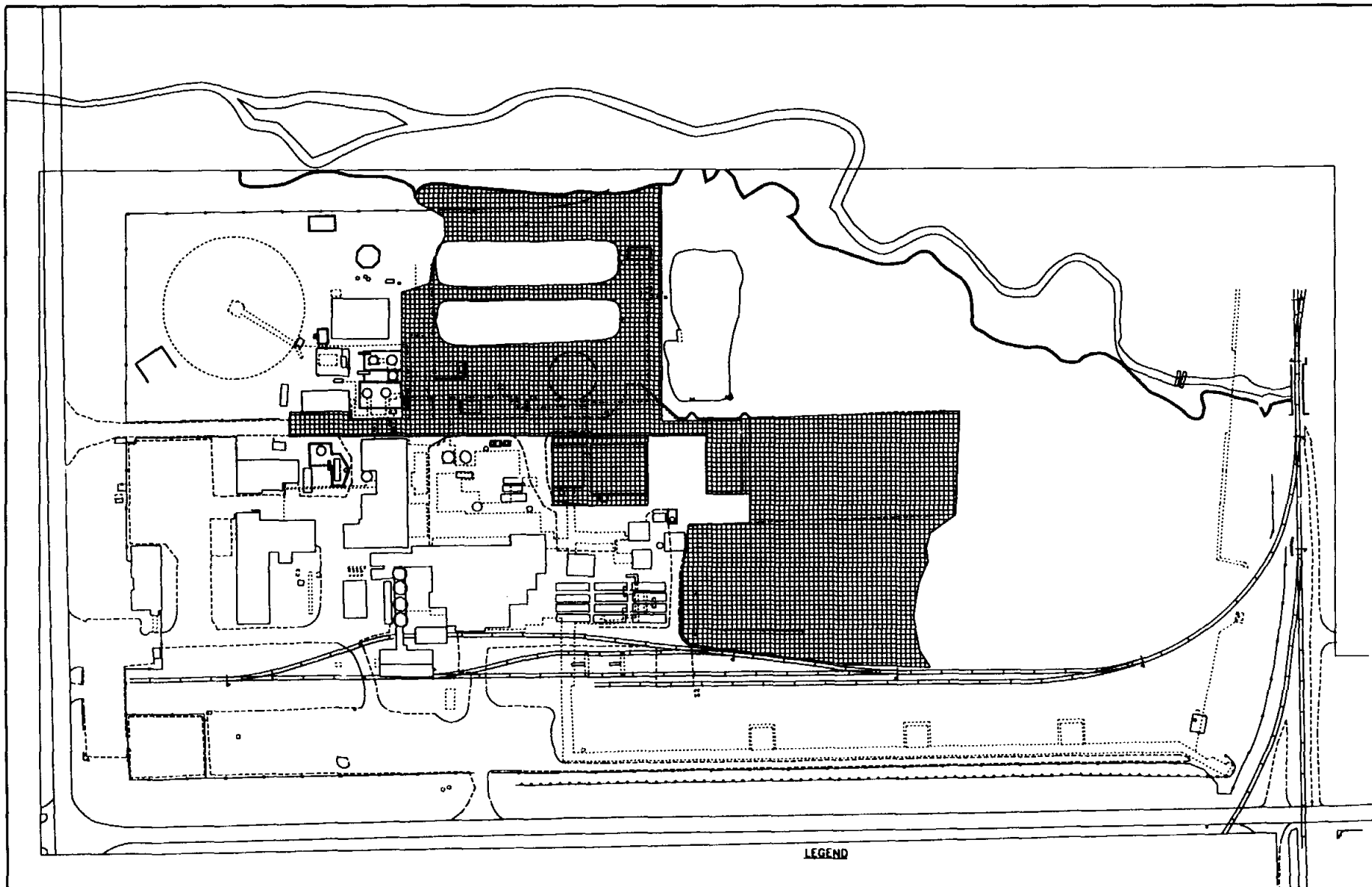
TABLE 2-1. DELINEATION SAMPLING PROGRAM

BORING ID	BORING DEPTH (ft)	NUMBER OF SAMPLES
Z1	surface	1
Z2	surface	1
Z3	7	1 (0-2)
Z4	7	1 (0-2)
Z5	14	4
Z6	4	1 (1-3)
Z7	surface	1
Z8	surface	1
Z9	4	1 (0-2)
Z10	4	1 (2-4)
Z11	surface	1
Z12	surface	1
Z13	surface	1
Z14	surface	1
Z15	12	3
Z16	12	3
R1	surface	1
R2	surface	1
R3	surface	1
<b>Total Number of Samples</b>		310
<b>Number of Duplicates (1 per 20 samples)</b>		16
<b>Number of Matrix Spike/Matrix Spike Duplicates (1 per 20 samples)</b>		16
<b>Number of Surface Sampling Locations</b>		15
<b>Number of Drilling Locations</b>		47
<b>Total Drilling Depth (ft)</b>		624
<b>Average Drilling Depth (ft)</b>		13

NOTES:

- 1) X are samples in the 50-foot grid sampling plan.
- 2) Z are samples in the 100-foot grid sampling plan.
- 3) R are random samples outside the 100-foot grid sampling plan.
- 4) Samples in the 100-foot grid are taken in the upper 2 feet for every 4-foot vertical interval.
- 5) The parenthesis represents the depth the sample will be taken for that location.
- 6) Boring depths are based on existing sample data or assumed at an average drilling depth of 14 feet.



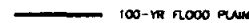


LEGEND



AREAS ASSOCIATED OR POTENTIALLY ASSOCIATED WITH INCIDENTAL VEHICULAR MOVEMENT OF PCBs

SCALE: 1" = 60'  
0 60 120 ft



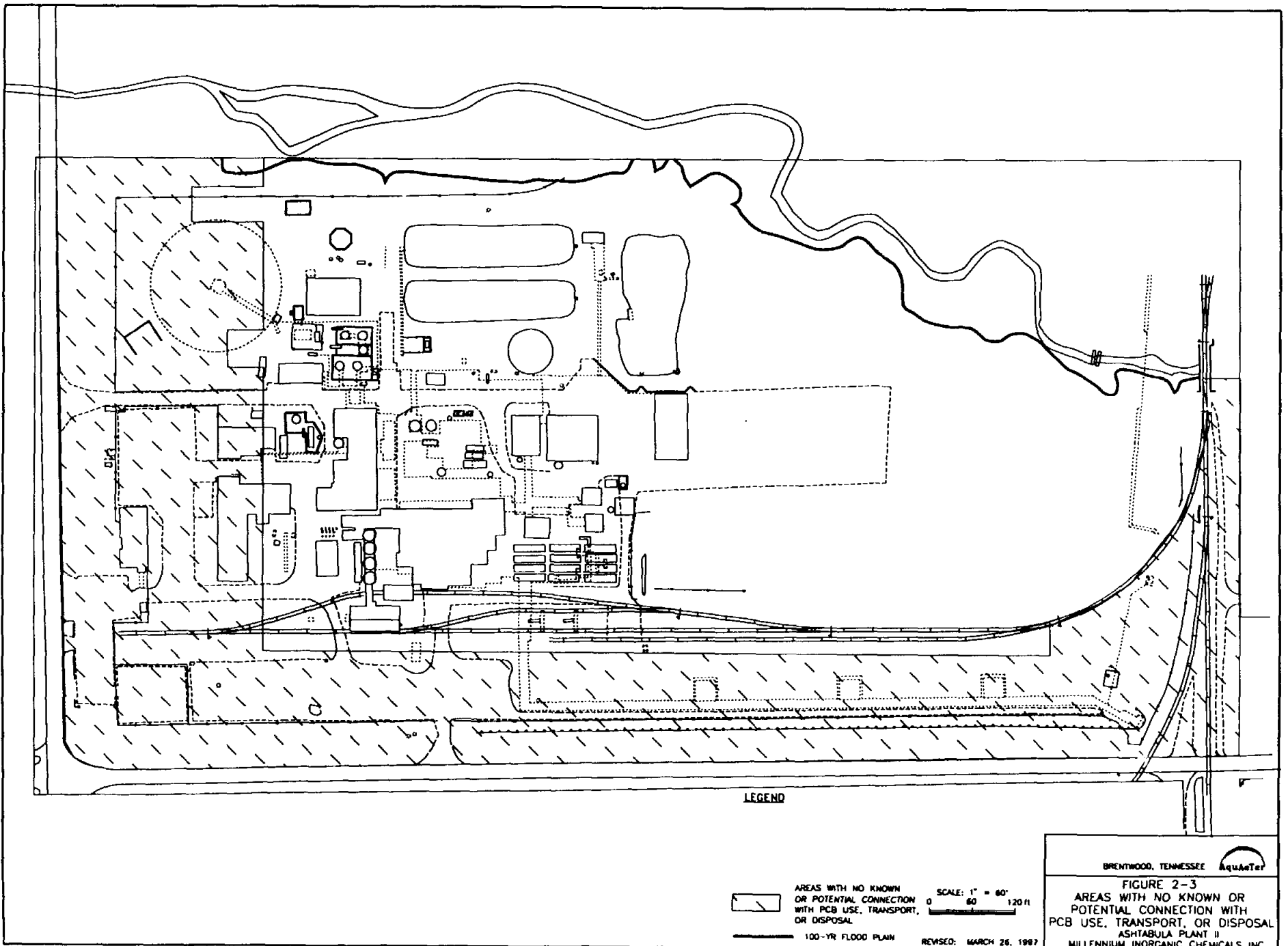
100-YR FLOOD PLAIN

REVISED: MARCH 28, 1997



BRENTWOOD, TENNESSEE




FIGURE 2-2  
AREAS ASSOCIATED OR POTENTIALLY ASSOCIATED WITH INCIDENTAL VEHICULAR MOVEMENT OF PCBs  
ASHTABULA PLANT II  
MILLENNIUM INORGANIC CHEMICALS INC.



LEGEND

 AREAS WITH NO KNOWN OR POTENTIAL CONNECTION WITH PCB USE, TRANSPORT, OR DISPOSAL  
 100-YR FLOOD PLAIN  
 SCALE: 1" = 60'  
 0 60 120 ft  
 REVISED: MARCH 26, 1997

BRENTWOOD, TENNESSEE   
 FIGURE 2-3  
 AREAS WITH NO KNOWN OR POTENTIAL CONNECTION WITH PCB USE, TRANSPORT, OR DISPOSAL  
 ASHTABULA PLANT II  
 MILLENNIUM INORGANIC CHEMICALS INC.

**FIGURE 2-4**  
**DELINEATION SAMPLING PROGRAM**



## SECTION 3

### LABORATORY ANALYSIS

PCBs are the parameter of concern for this site. All samples will be analyzed for PCBs, specifically Aroclor 1016, Aroclor 1221, Aroclor 1232, Aroclor 1242, Aroclor 1248, Aroclor 1254, and Aroclor 1260. The samples will be analyzed using Method 8081 as described in Test Methods for Evaluating Solid Waste, third edition and subsequent revisions (SW846). The Lancaster Laboratories, Inc. Standard Operating Procedures (SOP) for this method is presented in Appendix 1.

The quality assurance/quality control (QA/QC) procedures to be followed will be similar to those outlined by Woodward-Clyde Consultants (WCC) in the "Source Control Operable Unit RI/FS Revised QAPjP and Field Sampling Plan, Phase I" (December 1992) and the "Phase III Floodplain Sampling Design Investigation Quality Assurance Project Plan Addendum" (November 8, 1994). While the referenced plans are specific to WCC sampling events, the premise of these reports will be used for this sampling event. The sampling and oversight will be performed by **AquAeTer** and the laboratory analysis will be performed by Lancaster Laboratories, Inc. Lancaster Laboratories has experience working with the Contract Laboratory Program (CLP).







- ◆ Sample Number/Location 01, 02....n, n = number of samples in the Matrix
- ◆ Sample Type S = Soil, D = Duplicate, M = Matrix Spike, Duplicate

## **SAMPLE COLLECTION AND QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)**

### Surface Soil Samples

Surface soil samples will be collected at the marked sampling location. Field personnel will describe and photograph the sampling location using a white board to identify the sample point in the photograph. All sampling data will be entered into a bound field log book. Specific data will be recorded on water-resistant data sheets (i.e., GPS grid location descriptions) and kept in the field in a three-ring binder or other appropriate holder. Any variation from the procedures outlined in this DSP will be recorded in a field variance notebook.

Surface samples will be collected in the following manner:

- ◆ Soil samples will be collected from the upper 6 inches of soil after existing cover materials (i.e., vegetation, gravel, concrete, or asphalt) are removed from the sampling location. The sampling area will be approximately 1 foot by 1 foot.
- ◆ A sufficient amount of soil will be collected for the PCB analysis and placed in the appropriate laboratory containers.
- ◆ Duplicate and Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples will be collected by distributing soil equally into two sets of sample containers at a frequency of 1 per 20 samples collected, as described in the Sediment Operable Unit Quality Assurance Project Plan (SOU QAPjP). MS/MSD samples will be prepared by the laboratory from the environmental samples collected by the field personnel.

These samples will be analyzed for PCBs to evaluate whether matrix spike recoveries falling outside the acceptable windows are attributable to sample matrix interferences or to laboratory analytical errors.

- ◆ In order to minimize cross contamination between sample locations, any equipment or personal protective equipment which potentially comes into contact with contaminated material will be changed or decontaminated between sampling events.
- ◆ Surface soil samples will be analyzed for PCBs (Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260). Sample containers will be stored in iced, insulated coolers with appropriate chain-of-custody documentation. Samples will be sent to the laboratory via overnight carrier.
- ◆ The minimum sample size for each analysis requested is 50 grams. Therefore, each sample will be placed in a 4-ounce wide-mouth glass jar with a Teflon lining for shipment to the analytical laboratory.

Collection of field blanks and inclusion of trip blanks in sample shipments is not required for soil samples.

### Subsurface Samples

Subsurface soil samples will be collected at the marked sampling locations. Field personnel will describe and photograph the sampling location using a white board to identify the sample point in the photograph. Sampling data recorded in the field will be entered into a bound field log book. Specific data will be recorded on water-resistant data sheets (i.e., boring logs, GPS grid location descriptions) and kept in the field in a three-ring binder or other appropriate holder. Any variation

from the procedures outlined in this Delineation Sampling Program will be recorded in a field variance notebook.

Subsurface samples will be collected in the following manner:

- ◆ The drill rig auger will be advanced after existing cover materials (i.e., vegetation, gravel, concrete, or asphalt) are removed from the sampling location.
- ◆ A 2-foot split spoon sampler will be used and boring logs will be maintained over the entire drilling depth. When the DSP specifies that samples are to be collected at 4-foot vertical intervals, the samples will be collected in the upper two feet of each interval. For example, in a 12 foot deep boring, samples would be collected at 0 to 2 feet, 4 to 6 feet, 8 to 10 feet, etc.
- ◆ A sufficient amount of soil will be collected for the PCB analysis. The soil will be placed in the appropriate laboratory containers.
- ◆ Duplicate and MS/MSD samples will be collected by distributing soil equally into two sets of sample containers at a frequency of 1 per 20 samples collected, as described in the SOU QAPjP. MS/MSD samples will be prepared by the laboratory from the environmental samples collected by the field personnel. These samples will be analyzed for PCBs to evaluate whether matrix spike recoveries falling outside the acceptable windows are attributable to sample matrix interferences or to laboratory analytical errors.
- ◆ In order to minimize cross contamination between sample locations, any equipment or personal protective equipment which has the potential to cause cross-contamination will be changed or decontaminated between sampling events.

- ◆ Surface soil samples will be analyzed for PCBs (Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260). Sample containers will be stored in iced, insulated coolers with appropriate chain-of-custody documentation. Samples will be sent to the laboratory via overnight carrier.
- ◆ The minimum sample size for each analysis requested is 50 grams. Therefore, each sample will be placed in a 4-ounce wide mouth glass jar with a Teflon lining for shipment to the analytical laboratory.

Collection of field blanks and inclusion of trip blanks in sample shipments is not required for soil samples.

## **DECONTAMINATION**

Decontamination of personnel and equipment will be performed to prevent possible cross contamination and transport of contaminants off-site or between work areas. A mobile decontamination station will be established near each sample location.

### Personnel Decontamination

Sampling personnel will be required to use new, clean gloves while collecting each sample. Non-disposable personal protective gear will be decontaminated before personnel exit the hot zone and at the end of each day. The personnel decontamination procedure to be performed when personnel exit the hot zone and at the end of each day is as follows:

1. Place equipment and/or samples in designated area;
2. Remove outer coveralls and booties and place in plastic bags;

3. Wash boots and outer gloves using soap (Alconox or equivalent), and potable water rinse. Place gloves and disposable overboots in plastic bags;
4. Remove respirator, if used, sanitize, and store in appropriate place;
5. Wash hands and face;
6. Collect and store disposable equipment for disposal; and
7. Collect and store rinseate for disposal.

#### Sampling Equipment Decontamination

Whenever possible, sampling equipment will be dedicated and thus will not require decontamination. However, for non-dedicated equipment, the following decontamination procedure described below will be followed.

Sampling equipment will be decontaminated before use. Reusable, non-dedicated equipment will be decontaminated between each sampling event and before removal from the exclusion zone.

The procedure for sampling equipment decontamination is as follows:

1. Remove loose soil by wiping with a paper towel wetted in cleaning solution;
2. Wash with Alconox or other low-phosphate detergent wash;
3. Rinse with organic-free deionized (DI) water;
4. Rinse with isopropanol, methanol, or hexane;
5. Rinse with DI water;
6. Allow to air dry;
7. Triple rinse with DI water; and
8. Collect and store rinseate for disposal.



## **SECTION 5**

### **SCHEDULE**

The described field activities will be initiated upon Agency approval of this plan. It is anticipated that the field activities will be conducted in the Spring of 1997.





**APPENDIX 1**

**LANCASTER LABORATORIES, INC.  
STANDARD OPERATING PROCEDURE  
SW846 METHOD 8081**



Analysis #1216, 1224, 1225, 1363,  
 0819, 1866, 1867, 5367,  
 4354, 6000, 6001, 6005,  
 6624, 6677, 6678

Initiated Date: 11/04/87

Effective Date: JUL 17 1995

**Analysis of Soil and Sediment for Pesticides and PCBs**

**Reference:**

Methods 3540B, 3550A, 8081, 8141A, *Test Methods for Evaluating Solid Waste*, SW-846, September 1994.

**Scope:**

This method is applicable to the measurement of the following organochlorine and organophosphorus pesticides and PCBs in soil and sediment samples:

<u>Compound</u>	<u>Limit of Quantitation (mg/kg)</u>
Kepone	0.7
alpha-BHC	0.01
beta-BHC	0.01
delta-BHC	0.01
gamma-BHC (Lindane)	0.01
Heptachlor	0.01
Aldrin	0.01
Heptachlor epoxide	0.01
Endosulfan I	0.01
Dieldrin	0.01
Atrachlor	0.5
Atrazine	0.1
Metolachlor	0.5
Cyanazine	0.1
Simazine	0.2
4,4-DDE	0.01

Analysis #1216, 1224, 1225, 1363,  
 0819, 1866, 1867, 5367,  
 4854, 6000, 6001, 6005,  
 6624, 6677, 6678

Initiated Date: 11/04/87  
 Effective Date: JUL 17 1995  
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<u>Compound</u>	<u>Limit of Quantitation</u> <u>(mg/kg)</u>
Endrin	0.01
Endosulfan II	0.01
4,4-DDD	0.01
Endosulfan sulfate	0.03
4,4-DDT	0.01
Endrin aldehyde	0.01
Methoxychlor	0.05
Chlordane, technical	0.05
Toxaphene	2.0
PCB-1016	0.2
PCB-1221	0.2
PCB-1232	0.2
PCB-1242	0.2
PCB-1248	0.2
PCB-1254	0.2
PCB-1260	0.2
Ronnel	0.01 (.1 for 6678)
o,p-DDE	0.01
o,p-DDD	0.01
o,p-DDT	0.01
Mirex	0.01
Methyl parathion	0.02
Ethyl parathion	0.02
Diazinon	0.01 (.04 for 6678)
Malathion	0.05
Triphos	0.02
Trithion	0.05
HCB	0.01
Telodrin	0.01
Disulfoton	0.1
Thimet (phorate)	0.1
Famphur	0.1
alpha-chlordane	0.01

Analysis #1216, 1224, 1225, 1363,  
 0819, 1866, 1867, 5367,  
 4854, 6000, 6001, 6005,  
 6624, 6677, 6678

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Effective Date: JUL 17 1995

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<u>Compound</u>	<u>Limit of Quantitation</u> (mg/kg)
gamma-chlordane	0.01
Endrin ketone	0.1
Hexachlorophene	10.0
Dichlorvos	.020
Mevinphos	.030
Demeton-O	.040
Ethoprop	.040
Naled	.100
Demeton-S	.030
Fenthion	.040
Dursban (Chlorpyrifos)	.050
Trichloronate	.100
Merphos	.500
Stirophos	.100
Tokuthion	.050
Fensulfothion	.200
Bolstar	.050
Guthion (Azinphos-methyl)	.060
Coumaphos	.500

The extraction procedure requires 1 to 2 hours per sample using sonic probe. One technician can prepare eight samples in an 8-hour day. Soxhlet extraction takes 16 hours, followed by 1 to 2 hours per sample. Each sample extract takes 40 minutes to chromatograph and may require further cleanup by florisil, sulfuric acid treatment, TBA treatment, or dilution if interferents such as oxygenated organics, unsaturated organics, or elemental sulfur are present. Refer to Appendices II, VI, and VII for details on each cleanup procedure.

This method is used for analyzing soil and sediment samples scheduled for Lancaster Laboratories Analyses #1224, 1225, 1216, 1363, 1866, 1867, 5387, 4854, 6678, and 6624. Analysis #6000, 6001, and 6005 are for a specific

Analysis #1216, 1224, 1225, 1363,  
0819, 1866, 1867, 5367,  
4854, 6000, 6001, 6005,  
6624, 6677, 6678

Initiated Date: 11/04/87

Effective Date:

JUL 17 1995

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client's work only to meet contractual obligations. They are identical to existing scans except they are for pesticides only—no PCBs are included (6000 = 1225; 6001 = 1866; 6005 = 1224). Scan #6624 has LOQs equal to CRLs listed in CLP.

Sonic probe extraction is the primary extraction technique. Nonsoil solid samples such as paperboard, garbage, and textiles will be extracted using soxhlet. All samples for organophosphate pesticides (6678, 1867, 5367) will also be extracted using soxhlet as sonic probe is not an option for method 8141A.

#### Basic Principles:

A 30-g portion of homogenized sample is dried with sodium sulfate and extracted with 50% methylene chloride in acetone. The extract is filtered, dried, concentrated by evaporation, and put through Florisil. The pesticides and PCBs are then identified and quantitated using gas chromatography. Sulfuric acid treatment, TBA treatment, or dilution can be used to eliminate matrix interferences which introduce large, unresolvable peaks in the chromatogram.

#### Apparatus:

1. Beakers - 250 mL (glass or stainless steel)
2. Glass stirring rods
3. Buchner funnel
4. Erlenmeyer filter flask - 500 mL
5. Kuderna-Danish concentrator flasks - 500 mL with 10 mL graduated concentrator tubes

Analysis #1216, 1224, 1225, 1363,  
0819, 1866, 1867, 5367,  
4854, 6000, 6001, 6005,  
6624, 6677, 6678

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6. Three-ball Snyder columns
7.  $\text{Na}_2\text{SO}_4$  drying columns - 29 mm x 200 mm
8. Glass wool
9. Steam bath
10. Glass beads
11. Screw-cap vial - 12-mL capacity
12. Ultrasonic cell disruptor, heat systems - Ultrasonics, Inc. Model #W-385, or equivalent
13. Round-bottom flasks - 500 mL with 24/40 F joint or equivalent
14. Soxhlet extraction tube - With 24/40 m bottom joint and 55/50F top joint or equivalent
15. Allihn condenser - With 55/50 m bottom joint or equivalent
16. Soxhlet extraction thimbles
17. Boiling chips
18. Heating mantles
19. Cool-flow

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20. HP5890 gas chromatograph - Fitted with electron capture detector, or equivalent, for Analysis #1216, 1224, 1225, 1866, 1363, 4854, 6000, 6001, 6005, and 6624
21. Varian 3600 gas chromatograph - Fitted with a nitrogen-phosphorous detector, or equivalent, for Analysis #1867, 5367, and 6678
22. DB608 megabore column - 30 m x .53 mm x 0.83  $\mu$ m or equivalent
23. DB1701 megabore column - 30 m x .53 mm x 1.0  $\mu$ m or equivalent
24. DB210 megabore column - 30 m x .53 mm x 1.0  $\mu$ m or equivalent
25. Integrating system (like Chrom Perfect by Justice Innovations or equivalent)
26. Autosampler vials

Reagents:

1. Hexane - Pesticide grade
2. Methylene chloride - Pesticide grade
3. Acetone - Pesticide grade
4. Sodium sulfate - Baked in a muffle furnace for 4 hours at 400°C; store in a glass jar labeled with preparation and expiration dates (1 year from preparation)
5. UPC Nitrogen



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6. Pesticide standards, prepared as listed in SOP-PP-021, "Standards Preparation, Coding and Storage," and SOP-PP-003, "Standards Traceability and Monitoring."

#### Safety Precautions:

Avoid inhaling the solvents or getting them on the skin. Wear gloves when handling methylene chloride. To protect the ears from the noise of the cell disruptor horn, the unit should be placed in a sound reduction box. All concentrations are done in a fume hood. Read all lab notes to target potentially hazardous samples. Check all Soxhlet glassware for cracks to ensure no solvent leaks into heating mantles. Be sure cool flow apparatus is on and condensers are cold before starting the extraction. Do not use wet mantles—discontinue analysis if a spill or leak onto a mantle occurs.

#### Sample Preservation and Holding Times:

The holding time for samples is 14 days from collection. Samples are not preserved: each sample is homogenized upon submission to our lab and then stored at  $4^{\circ} \pm 2^{\circ}\text{C}$ .

#### Sonic Probe Extraction:

1. Weigh out  $30 \pm .05$  g of sample into a 250-mL beaker.
2. Add at least 60 g of anhydrous powdered sodium sulfate and mix well. Extra sodium sulfate should be added until a free-flowing mixture is obtained.
3. If the sample is a wipe, remove it from the vial with clean tweezers and place it in a beaker. Do not add  $\text{Na}_2\text{SO}_4$ . Rinse the vial with a few mL of acetone/methylene chloride and add to the beaker. Any solvent in the vial is also added to the beaker for extraction.

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4. Add matrix spikes where applicable.
5. Add appropriate surrogate standards.
6. Add 100 mL of 50% methylene chloride in acetone to the sample.
7. Rinse the cell disrupter horn with deionized water, followed by acetone. Then place a beaker with acetone under the horn and run through one sonication cycle. Be sure horn is clean before processing samples.
8. Place the beaker with the sample under the disrupter horn of the sonicator so that the tip of the horn is 1/4 inch below the surface of the solvent, but above the sediment layer.
9. Sonicate for three minutes with the percent duty cycle at 50% and the cycle at 1-second pulse.
10. Decant and filter extract into a Buchner funnel through Whatman #3 filter paper using vacuum filtration by thoroughly wetting the filter paper with a portion of the 50% solution, then decanting the extract onto the center of the paper to keep small particulates from going under the edge of the paper. Then rinse the filter paper with a small amount of 50% solution.
11. Repeat extraction two more times with two additional 100-mL portions of 50% solution. Before each sonication, make sure sodium sulfate is free flowing. If not, break up any lumps with a glass stirring rod. Decant and filter the solvent after each sonication. After the final sonication, pour off all the liquid portion, including any suspended particulate matter.
12. Add 50 to 100 mL of 50% solution to the beaker and rinse the soil and beaker. Add this to the funnel. Rinse the Buchner funnel one more time.

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13. If, at this point, the filtrate still contains particulate matter, refilter through a clean piece of #3 paper.
14. Transfer the final filtrate into a K-D flask with a 10-ml concentrator tube. Rinse Erlenmeyer and put rinse into the K-D. Proceed to Concentration Procedure.

#### Soxhlet Procedure:

1. Put 300 mL of 50% methylene chloride in acetone into a 500-mL round-bottom flask. Add a few boiling chips and place on a heating mantle.
2. Weigh  $30 \pm .05$  g of homogenized solid sample into an extraction thimble. Less can be used if the material is very light and 30 g will not fit into a thimble. Add spiking solution directly onto solid in thimble if sample is a spike; add surrogate in the same manner to all samples and QC. Place the thimble in a soxhlet extraction tube and assemble soxhlet setup. Be sure all joints are tight. If the sample is a wipe, remove it from the vial with clean tweezers and place it in a thimble. Rinse the vial with a few mL of 50% solution and add to the round-bottom flask.
3. Turn on cool-flow and, when condensers are cold, turn on heating mantles. Adjust the heating rate so that the chamber empties once every 10 to 15 minutes (approximately #5 to 6½ on temperature regulator). When solvent starts collecting in the extraction tube, check all joints and the arms on the extraction tube for leaks. Replace broken extraction tubes, tighten joints, or restart with new glassware to correct any problems.
4. Allow extraction process to continue for 16 hours. Periodically check the apparatus to ensure solvent is condensing and extractors are emptying properly.

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5. Turn off the heating mantles and cool the apparatus. Turn off cool flow and disassemble extractor. Pour any solvent remaining in the extraction tube into the round-bottom flask. Discard the thimble containing the sample.
6. Assemble a Kuderna-Danish (K-D) apparatus by attaching a 10-mL concentrator tube to a 500-mL flask using Teflon tape and a plastic clip to secure the joint. Place a drying column on top which contains approximately 3 inches of  $\text{Na}_2\text{SO}_4$ .
7. Drain the extract through the drying column into the K-D. Rinse the round-bottom with approximately 20 mL of 50% and put this through the  $\text{Na}_2\text{SO}_4$  column into the K-D. Proceed to Concentration Procedure.

#### Concentration Procedure:

1. Add a boiling bead to the K-D. Attach a 3-ball Snyder column and prewet with methylene chloride and concentrate to approximately 1 mL on a steam bath. Do not allow ampule to go dry.
2. Add 50 mL of hexane directly to the K-D through the Snyder column and concentrate to approximately 1 mL. Add another 50 mL hexane and concentrate again to 1 mL.
3. Cool, remove concentrator tube and adjust final volume to exactly 10 mL with hexane. Mix thoroughly.
4. If the sample is scheduled for Analysis #1867, 6678, or 5367, make a 10-fold dilution of the unflorisilied extract. Bottle this in a vial to run on the GC.
5. If the sample is scheduled for Analysis #1216, treat the 10-mL extract with acid as described in Appendix II.

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6. Florisil the acid-treated solution for Analysis #1216, the extract from Step 3 above for Analysis #1224, 1225, 1866, 6624, and 1363, as described in Appendix VI, Florisil Cartridge Cleanup. Store remainder of extract in a screw-cap vial in the freezer.

#### Gas Chromatographic Analysis:

Instrument setup for ECD (Analysis #1216, 1224, 1225, 1363, 1866, 6624, 4854, 6000, 6001, and 6005):

Detector - ECD

Detector temperature - 300°C

Oven temperature - 140°C, 10°C/min, 170°C, no hold, 3°C/min to 260°C, hold till all analytes elute

Carrier gas - He at 5 mL/min

Makeup gas - N<sub>2</sub> at 30 mL/min for Varian ECDs, 55 mL/min for H-P ECDs.

Injection size - 2 µL, direct injection

Injector temperature - 250°C

The conditions listed above are optimum but may vary as the linearity, sensitivity, and chromatography are improved on each GC System.

Instrument setup for nitrogen-phosphorus (Analysis #1867, 6678, and 5367):

Detector - nitrogen-phosphorus (thermionic specific)

Detector temperature - 300°C

Oven temperature - 140° to 270°C, 5°C/min, hold till all analytes elute

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Carrier gas - He @ 5 mL/min

Makeup gas - N<sub>2</sub> @ 30 mL/min

Injection size - 2  $\mu$ L, direct injection

Injector temperature - 280°C

The conditions listed above are optimum but may vary as the linearity, sensitivity, and chromatography are improved on each GC System.

1. Inject all standards of interest, as prepared following SOP-PP-021 and SOP-PP-003.
2. Inject the extract onto the GC columns).
3. Using a three-standard retention retention time window, compare retention times of peaks found in the sample chromatogram with those of the standards. Quantitate results which confirm on both columns.
4. If significant interference is present, treat with sulfuric acid as described in Appendix II, treat with TBA as described in Appendix VII and/or dilute the extract. Be sure cleanup is appropriate for the analytes of interest.
5. A more in-depth explanation of the GC setup and requirements for the data can be found in SOP-PP-007, "Setting Up and Checking an Analytical Sequence for Samples Analyzed for Polychlorinated Biphenyls (PCBs) by Method 8081, SW-846 or EPA Method 608," SOP-PP-009, "Setting Up and Checking an Analytical Sequence for Samples Analyzed by Method 8081, SW-846," and SOP-PP-010, "Setting Up and Checking an Analytical Sequence for Organophosphates by Method 8141A, SW-846."

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Calculations:

A. Single-component analytes

A five-point calibration curve is constructed for each single-component analyte. The results are calculated from this when the %RSD > 20%. Otherwise, the average response factor is used.

The calculations performed by the data system are:

1. Curve:

$$\text{As Received result in mg/kg} = \text{Extract conc.} \times \text{FV} \times \text{AF} \times \frac{\text{DF}}{\text{IW}}$$

Where:

$$\text{Extract conc. (}\mu\text{g/ml)} = \frac{\text{Peak height} - \text{Y-Intercept}}{\text{Slope}}$$

FV = Final volume of extract after florisil

AF = Additional factor

$$\text{AF} = \frac{\text{Total volume before florisil (10 mL)}}{\text{Volume removed for florisil cleanup}}$$

DF = Dilution factor

IW = Initial weight of sample extracted

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2. Average RF:

$$\frac{\text{Peak height of sample}}{ARF} \times FV \times AF \times \frac{DF}{IW}$$

Where:

$$RF = \frac{\text{Peak height of analyte}}{\text{Concentration of analyte in standard } (\mu\text{g/mL})}$$

ARF = Average response factor = the average of the RF for each level in the calibration.

Ex:

$$\text{Average RF} = \frac{RF \text{ Callb 1} + \dots + RF \text{ Callb 5}}{5}$$

3. A single-point calculation may be performed using a particular calibration level by substituting that RF into the equation for ARF.

B. Multiple-component analytes

The residues are identified by matching the retention times of the peaks in the sample to the peaks in the standard, as well as the peak pattern. The relative heights of the various peaks will help in identifying different isomers.

The peak heights generated by the integration system are used to calculate response factors for aroclors 1016, 1221, 1232, 1248, 1242, 1260, and 1254, technical chlordane, pyrethrins, and toxaphene.

$$\text{Response factor (RF)} = \frac{\text{Standard peak height}}{\text{Standard concentration (ppm)}}$$



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Sample concentrations are calculated by averaging the result from each peak chosen for quantitation. The quantitation of each peak is as follows:

$$\frac{\text{Sample height}}{RF} \times \frac{FV}{IV} \times AF \times DF = \text{mg/L as received}$$

#### Quality Assurance:

At least one reagent blank, and a reagent blank spike (laboratory control spike, LCS) are analyzed with every group of up to 20 samples. A spiked background soil, and a spike duplicate are analyzed with every batch of samples. (A batch consists of a maximum of 20 samples and can be added to for 14 days.) See Table 2 for a list of the analytes spiked for each analysis. SOP-PP-002, "QC Data Acceptability and Corrective Action," details the QC acceptance criteria and corrective action. SOP-PP-025, "Monitoring QC Data Acceptance Limits," outlines how acceptance limits are established and monitored for trends in surrogate and spike recovery. Analysis #1867, 6078, and 5367 use 2-nitro-m-xylene as a surrogate standard. All other analyses use tetrachloro-m-xylene (TCMX) and decachlorobiphenyl as surrogates. SOP-PP-021 details the composition and preparation of all standards used for the analyses.

Each lot of florisil is checked for proper elution patterns and acceptable recovery as detailed in Appendix VI. Each lot of solvent is checked for cleanliness according to SOP-PP-018, "Standard Operating Procedure for Checking Solvent Lots for Acceptability."

12161224.W60  
071195

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Prepared by: *Jeff A. Ken*

Date: *7/13/95*

Approved by: *Dorothy M. Love*

Date: *7/17/95*

Approved by: *Kathy L. DeNunzio*

Date: *7/17/95*

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Table I

Summary of Analysis Numbers

<u>Lab Analysis Numbers:</u>	<u>1216</u>	<u>1363</u>	<u>1866</u>	<u>1224</u>	<u>1225/ 6624</u>
<u>Compounds</u>					
alpha - BHC		X	X	X	X
beta - BHC		X	X	X	X
delta - BHC		X	X	X	X
gamma - BHC (Lindane)		X	X	X	X
Heptachlor		X	X	X	X
Aldrin		X	X	X	X
Heptachlor epoxide		X	X	X	X
Endosulfan I		X	X	X	X
Dieldrin		X	X	X	X
Endosulfan II		X	X	X	X
4,4-DDE		X	X	X	X
Endrin		X	X	X	X
4,4-DDD		X	X	X	X
Endosulfan sulfate		X	X	X	X
4,4-DDT		X	X	X	X
Endrin aldehyde		X	X	X	X
Methoxychlor		X	X	X	X
Toxaphene		X	X	X	X
Technical Chlordane		X	X	X	
PCB - 1018	X	X	X	X	X
PCB - 1221	X	X	X	X	X
PCB - 1232	X	X	X	X	X
PCB - 1242	X	X	X	X	X
PCB - 1248	X	X	X	X	X
PCB - 1254	X	X	X	X	X
PCB - 1260	X	X	X	X	X
Kepone, hexachlorophene			X		

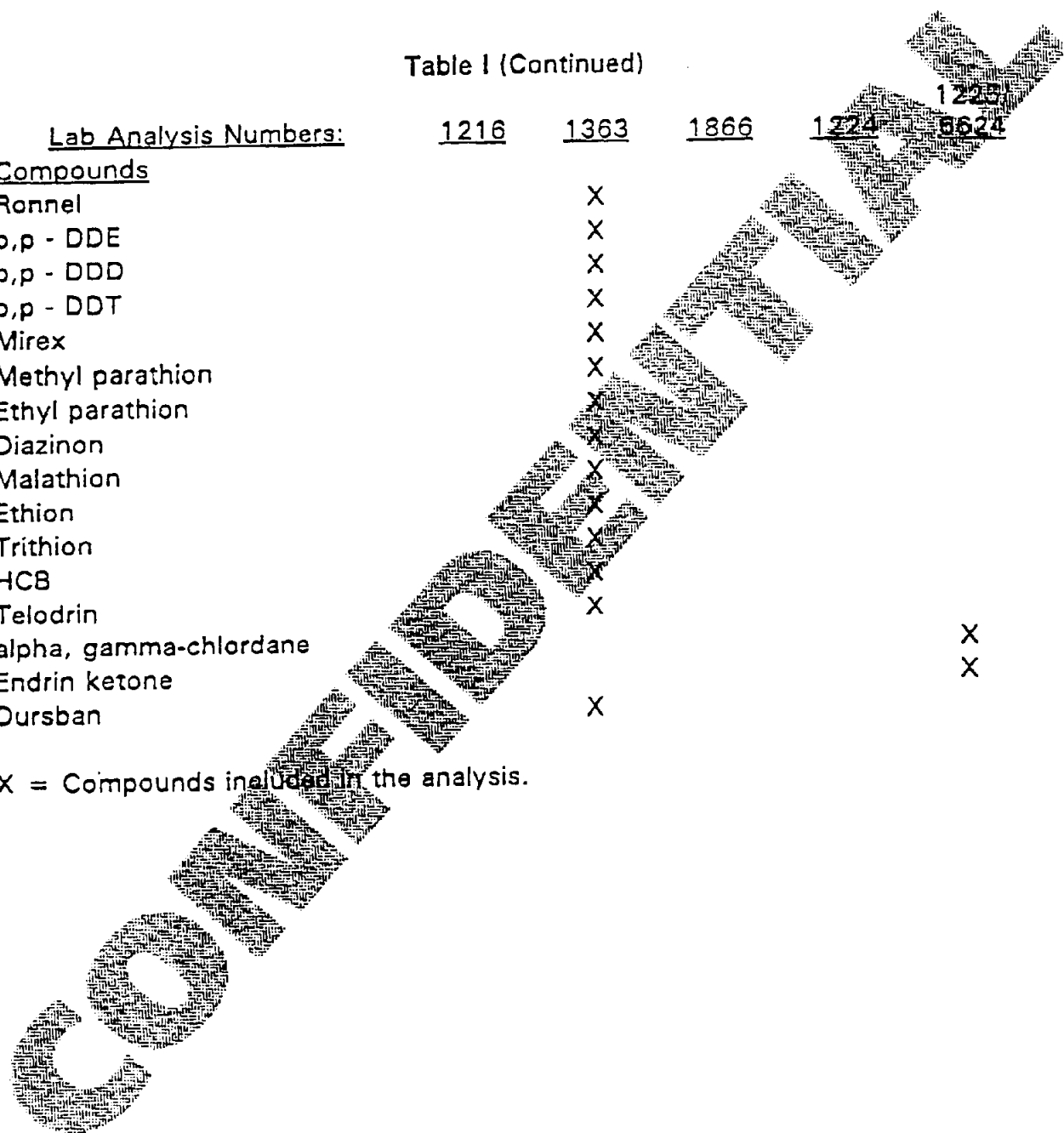
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<u>Lab Analysis Numbers:</u>	<u>1216</u>	<u>1363</u>	<u>1866</u>	<u>1224</u>	<u>1225</u> <u>6624</u>
<u>Compounds</u>					
Ronnel		X			
o,p - DDE		X			
o,p - DDD		X			
o,p - DDT		X			
Mirex		X			
Methyl parathion		X			
Ethyl parathion		X			
Diazinon		X			
Malathion		X			
Ethion		X			
Trithion		X			
HCB		X			
Telodrin		X			
alpha, gamma-chlordane					X
Endrin ketone					X
Dursban		X			

X = Compounds included in the analysis.



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<u>Lab Analysis Numbers</u>	<u>1867</u>	<u>5367</u>	<u>4854</u>	<u>6678</u>
<u>Compounds</u>				
Disulfoton	X			X
Phorate	X			X
Famphur	X			
Methyl parathion	X			X
Ethyl parathion	X			X
Alachlor		X		
Atrazine		X		
Metolachlor		X		
Cyanazine		X		
Simazine		X		
Pyrethrins			X	
Dichlorvos				X
Mevinphos				X
Demeton-O, S				X
Ethoprop				X
Naled				X
Fenthion				X
Dursban				X
Trichloronate				X
Merphos				X
Stirophos				X
Tokuthion				X
Fensulfotolon				X
Bofstar				X
Guthion				X
Coumaphos				X
Ronnel				X
Diazinon				X

X = Compounds included in the analysis.

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Table II

Summary of Analytes Contained in Spikes

<u>Lab Analysis Numbers:</u>	<u>1216</u>	<u>1363</u>	<u>1866</u>	<u>1224</u>	<u>1225/ 6624</u>
<u>Compounds</u>					
alpha - BHC		X	X	X	X
beta - BHC		X	X	X	X
delta - BHC		X	X	X	X
gamma - BHC (Lindane)		X	X	X	X
Heptachlor		X	X	X	X
Aldrin		X	X	X	X
Heptachlor epoxide		X	X	X	X
Endosulfan I		X	X	X	X
Dieldrin		X	X	X	X
Endosulfan II		X	X	X	X
4,4-DDE		X	X	X	X
Endrin		X	X	X	X
4,4-DDD		X	X	X	X
Endosulfan sulfate		X	X	X	X
4,4-DDT		X	X	X	X
Endrin aldehyde		X	X	X	X
Methoxychlor		X	X	X	X
Toxaphene					
Technical Chlordane					
PCB - 1016	X				
PCB - 1221					
PCB - 1250					
PCB - 1242					
PCB - 1248					
PCB - 1254					
PCB - 1260	X				
Kepone, hexachlorophene			X		

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<u>Lab Analysis Numbers:</u>	<u>1216</u>	<u>1363</u>	<u>1866</u>	<u>1224</u>	<u>1225</u> <u>6624</u>
<u>Compounds</u>					
Ronnel					
o,p - DDE					
o,p - DDD					
o,p - DDT					
Mirex					
Methyl parathion					
Ethyl parathion					
Diazinon					
Malathion					
Ethion					
Trithion					
HCB					
Telodrin					X
alpha, gamma-chlordane					X
Endrin ketone			X		
Dursban					

X = Compounds included in the analysis.

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<u>Lab Analysis Numbers</u>	<u>1867</u>	<u>5367</u>	<u>4854</u>	<u>6678</u>
<u>Compounds</u>				
Disulfoton	X			X
Phorate	X			X
Famphur	X			
Methyl parathion	X			X
Ethyl parathion	X			X
Alachlor		X		
Atrazine		X		
Metolachlor		X		
Cyanazine		X		
Simazine		X		
Pyrethrins			X	
Dichlorvos				X
Mevinphos				X
Demeton-O,S				X
Ethoprop				X
Naled				X
Fenthion				X
Dursban				X
Trichloronate				X
Merphos				X
Stirophos				X
Tokuthion				X
Fensulfothion				X
Bolstar				X
Guthion				X
Coumaphos				X
Ronnel				X
Diazinon				X

X = Compounds included in the analysis.