

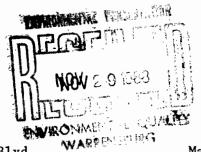
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Mahwah, NJ 07430

201 529-0800

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SOIL SAMPLING WORK PLAN CIBA-GEIGY MAIN PLANT SITE GLENS FALLS, NEW YORK

Prepared for:

CIBA-GEIGY Corporation Lower Warren Street Glens Falls, New York 12801

Prepared by:

AWARE Incorporated 1200 MacArthur Boulevard Mahwah, New Jersey 07430

November, 1988

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1.0 INTRODUCTION

This work plan for soil sampling and analysis has been prepared in response to the Preliminary Assessment, Main Plant Site Technical Notice of Deficiency (TNOD) issued by NYSDEC. The plan describes the objectives, technical approach and specific methods to be employed in the collection of soil samples at the CIBA-GEIGY main plant site in Glens Falls, New York. Although AWARE has been the principal author, this plan represents a joint effort on the part of several consultants to CIBA-GEIGY on this project. The plan makes specific reference and relies upon several documents which have not been reproduced for These include the Preliminary Assessment by Malcolm Pirnie this submittal. (September 1987) and the summary of manufacturing history prepared by CIBA-GEIGY (November 1988). Lastly, this work plan is but one of several work plans and ongoing studies being prepared and implemented by CIBA-GEIGY. Together these plans and studies comprise an overall remedial investigation program for the plant site.

1.1 OBJECTIVES AND SCOPE

The overall goal of this sampling plan is to provide the appropriate soil data necessary for the evaluation of remedial alternatives. In order to achieve this goal, the following objectives have been identified.

- Determine the extent that waste/fill deposits may have migrated off-site, particularly from the western portion of the site.
- Determine the distribution of contaminants in both the waste/fill deposits and naturally occurring soils.
- Evaluate the mobility or leachability of specific contaminants to the groundwater flow system.
- Provide additional data regarding the thickness and extent of natural clay deposits which may locally influence the direction of groundwater flow in the overburden.

 Provide additional data regarding the top of bedrock surface configuration.

The scope of this proposed investigation focuses on four specific concerns put forth in the Main Plant Site TNOD. These are summarized as follows:

- TNOD Item #1 Conduct a reconnaissance boring program to determine the limits of waste/fill areas.
- TNOD Item #3 Investigate source areas upgradient of known groundwater contamination.
- TNOD Item #4 Evaluate the industrial sewer as a potential source of soil contamination.
- TNOD Item #8 Investigate the quality of soils beneath floor slabs of on-site buildings.

This work plan also addresses the subsurface conditions of the north and south waste piles. Although this area is the subject of a separate TNOD dealing with the closure of these units, additional soils characterization is best achieved through this work plan. All other items contained in the Main Plant Site TNOD will be addressed under separate work plans.

1.2 PREVIOUS INVESTIGATIONS

An extensive preliminary soil investigation was conducted by Malcolm Pirnie in 1987. Between February 5 and April 3, 1987, Malcolm Pirnie and Dunn Geoscience Corporation completed the drilling of approximately 75 soil borings (Figure 1-1). A total of approximately 220 samples were collected for laboratory analysis of metals. Of these, approximately 54 were also analyzed for E.P. Toxicity Metals and approximately 157 samples were analyzed for organics. This program has been summarized on Table 1-1.

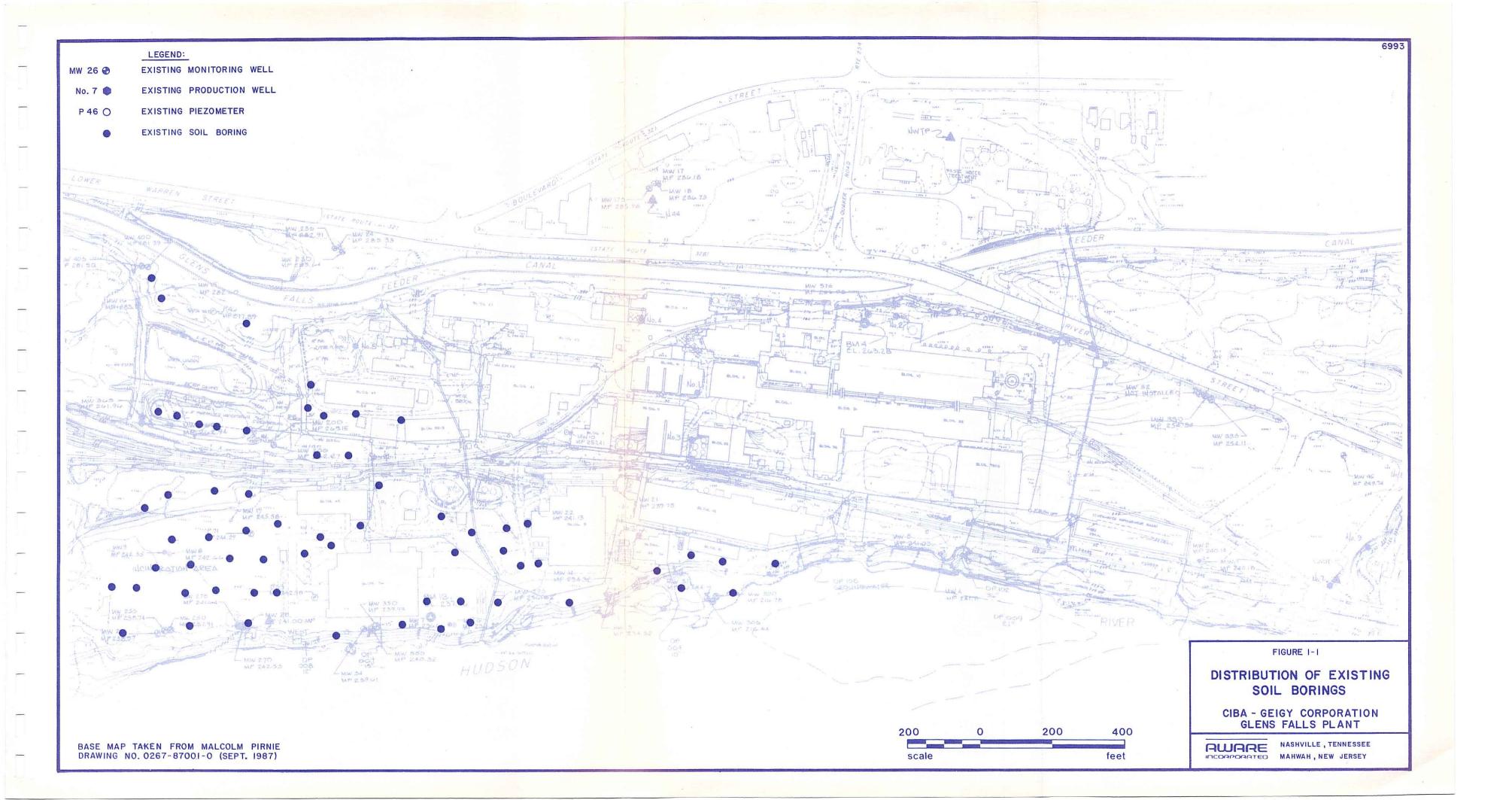


TABLE 1-1
SUMMARY OF MALCOLM PIRNIE SOIL INVESTIGATION

Area of Investigation	Numbe	er of Sampl	les per	Materia	al Type	
	Waste/Fill	Topsoil	Sand	Silt	Clay	Total
North Waste Pile	20	0	0	2	3	25
South Waste Pile	40	0	0	1	0	41
South/West Building 49	10	0	3	0	3	16
Incineration Area	18	0	17	0	6	41
Vicinity Building 56	35	0	19	1	12	67
South Building 45	3	3	7	1	3	17
Parking Lot	4	0	6	2	1	13
Total	130	3	5 2	7	28	220

Although the 1987 investigation provides a good deal of subsurface information, it does not fully achieve the objectives identified above. It is the intent of this work plan to build on the existing data and to fill in those areas which require more information.

2.0 TECHNICAL APPROACH

In order to achieve the objectives stated above, the following issues must be addressed:

- the spatial distribution of soil borings
- the depth interval(s) from which soil samples will be collected for laboratory analysis
- screening methodologies for metals and organics
- the chemical parameters which will be analyzed
- the leaching protocol to be used to evaluate the mobility of the contaminants

2.1 DISTRIBUTION OF BORINGS

Generally speaking, the selection of soil boring locations can be made based on either a biased or unbiased approach. In this work plan a combination of these two approaches has been developed. An unbiased grid has been established across the site in order to provide coverage of those areas which have not been investigated previously. These areas include the central and eastern portions of the plant.

A biased boring plan has also been developed to supplement the reconnaissance plan. This biased approach takes into consideration the results of the Preliminary Assessment and the operational history of the site. Specifically, these areas include:

The area to the west of the North Lagoon and associated waste piles, and the area to the west of the incineration area. The preliminary assessment did not address these off-site areas. Additional borings are needed to determine the extent of migration of site related waste/fill deposits.

- The area of the parking lot at which chrome ore was previously stockpiled by the U.S. Government. This area was investigated during the preliminary assessment, however, several additional borings are proposed to determine the extent of this ore.
- The area in the vicinity of the industrial sewer. The industrial sewer has, over the years, contained all of the chemical products and wastes from the plant. As a result, a series of borings have been located along its length.
- The soil beneath manufacturing areas. Most of the buildings on site were used to manufacture organic and inorganic pigments. Building slabs of specific buildings will be examined for likely pathways to the subsurface such as cracks, etc. Borings will be located at these points to determine if manufacturing operations may have contaminated the subsurface soils.

2.2 SAMPLE SCREENING AND ANALYSIS

In each boring all soil samples will be collected in two-foot depth increments from ground surface to refusal on bedrock. Each sample will be classified as fill, waste (tailings) or natural soil and sent to the laboratory for screening of metals and organics. The sample screening program includes use of a gas chromatograph (GC) for organics and the inductively coupled plasma (ICP) method for selected metals.

In the case of organics, a GC has been chosen over the more common field headspace method (HNU or OVA) for the following reasons. First, the GC will provide a hard copy chromatogram which will be available to document the results of the screening procedure. Secondly, the GC will detect semi-volatiles in addition to the volatile compounds, whereas the HNU would not. Finally, the implementation of sample screening in the controlled environment of a laboratory reduces instrument downtime and the field variability involved with using an HNU or OVA.

The sample screening data will be the primary factor in the selection of samples for more complete analysis. However, since the GC screening method provides only a qualitative response, a relatively strong complement of confirmatory analyses are proposed to support these results. For each boring in which a positive organic screening response is measured, two samples will be selected for laboratory analysis of volatile and semi-volatile organics. The sample associated with the highest response will always be chosen, as well as one other sample representing some lower (or non-detected) value. The objective will be to compile a range of screening values which have laboratory confirmation in order to correlate the remaining screening data.

Sample screening for metals by ICP is proposed in order to provide actual (documented) metal concentrations for each soil sample collected. This information will aid in the delineation of both the areal and vertical distribution of metals in the subsurface. The screening technique will use standard ICP methods for a "targeted" list of metals. In addition, samples from selected areas will be analyzed for "supplemental" inorganic parameters which were used in that area.

The metals screening process will provide quantitative data for the "targeted" list of metals. To ensure adequate coverage of other metals of concern, approximately five percent of the samples will be analyzed using ICP or AA methods as appropriate for an "expanded" list of metals. The target list, supplemental parameters and expanded list are further discussed below.

2.3 SELECTION OF PARAMETERS

Based on the historical account of site operations and the previous soil investigation program, four target metals have been identified for the metals screening procedure. These are barium, cadmium, chromium, and lead. The four metals will be analyzed by the ICP method for every sample collected during this investigation. In certain borings, each sample will also be analyzed for "supplemental" parameters as follows: Borings located in the vicinity of Buildings 42 and 49 will include analysis for mercury, whereas borings in the vicinity of Building 45 will include analysis for free and dissociable cyanide.

In order to provide adequate site-wide information on other metals, an "expanded" list of metals will be analyzed in approximately five percent of the total samples collected. This expanded list is identified on Table 2-1.

A similar account in terms of organic parameters indicates that both volatile and semi-volatile organics were used in some manufacturing operations. Thus the complete list of priority pollutant volatile and semi-volatile organics will be analyzed for those samples so designated.

2.4 SAMPLE LEACHING PROCEDURE

In order to reasonably simulate the in situ leachability of metals from the various waste and soil materials on site, a leaching test will be applied to selected samples. The test which has been chosen is ASTM D3987-85 (Annual Book of ASTM Standards, 1986): "Shake Extraction of Solid Waste with Water". In this procedure, 70 grams of sample is mixed with 1400 milliliters of water and agitated continuously for about eighteen hours. The extract is then passed through a 0.45 micron filter, pH measured, and analyzed for selected metals.

Unlike the E.P. Toxicity test, the water extraction simulates conditions in which the soil itself is the major factor in determining the pH of the extract. The target list of metals for analysis includes arsenic, barium, cadmium, chromium, cyanide, lead, mercury, and selenium. However, if other metals are present in significant concentrations in the soil, these will be included in the extract analysis.

Approximately 50 percent of the samples selected for the expanded metals analysis (approximately 25 samples) will be subjected to the leaching test. This selection will be primarily from the high and medium range total metal concentrations.

Table 2-1 Parameters for Soil Analysis

-							٠	
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Organics

Sample Screening (Target List)

Sample Screening

Barium Cadmium Chromium

Lead

Gas Chromatography

(PID and FID)

Supplemental List

Expanded List

Cyanide

Mercury

Volatiles (USEPA Method 8240)

Semi-volatiles (USEPA Method 8270)

Expanded List

Arsenic

Barium

Cadmium

Chromium

Cyanide

Strontium Iron Lead

Mercury

Selenium

Copper

3.0 FIELD INVESTIGATIONS

The implementation of this soil sampling work plan consists of 5 tasks as described below.

Task 1 - Site Reconnaissance

AWARE will stake and identify all boring locations designated in the work plan. A site reconnaissance will then be conducted over a one or two day period in order to discuss the logistics of implementing this work plan. It is anticipated that representatives of CIBA-GEIGY, Clement Associates, Aquatec Inc. and Empire Soils Investigations, Inc. will participate. The reconnassiance will include a site walkover to observe these boring locations and review their intended purpose. Access limitations, public health and safety concerns, and any other pertinent factors which come to light as a result of this walkover, will be discussed at this time.

Task 2 - Surface Geophysical Program

The application of non-disruptive surface geophysics may provide yet another tool with which to evaluate the extent of tailings and/or soils with high total metal concentrations. Based on the known site conditions, two surface geophysical methods have been identified as having the potential to provide this data. These are induced electromagnetics (EM) and earth resistivity. This task provides a means of evaluating the utility of each technique through an on-site trial survey.

The trial survey will be conducted along a predetermined line in an area of known subsurface conditions. Cross-section F-F' of the Preliminary Assessment Report, for example, contains a number of borings which depict tailings, natural soils and clean fill. This program will consist of one day of field evaluation for each technique and will include several runs using different instrument spacings.

The results will be presented in a brief report prepared within several weeks of the survey. The report will include all raw data and a comparative analysis of the different spacings. The report will also provide conclusions as to the utility of each method, limitations and the recommended approach for subsequent surveys (if appropriate).

If surface geophysics is demonstrated to be of value at the site, a work plan addendum will be prepared describing the proposed field program. The location of geophysical lines will be identified and the integration of these data with proposed borings will be discussed.

Task 3 - Soil Borings/Sample Collection

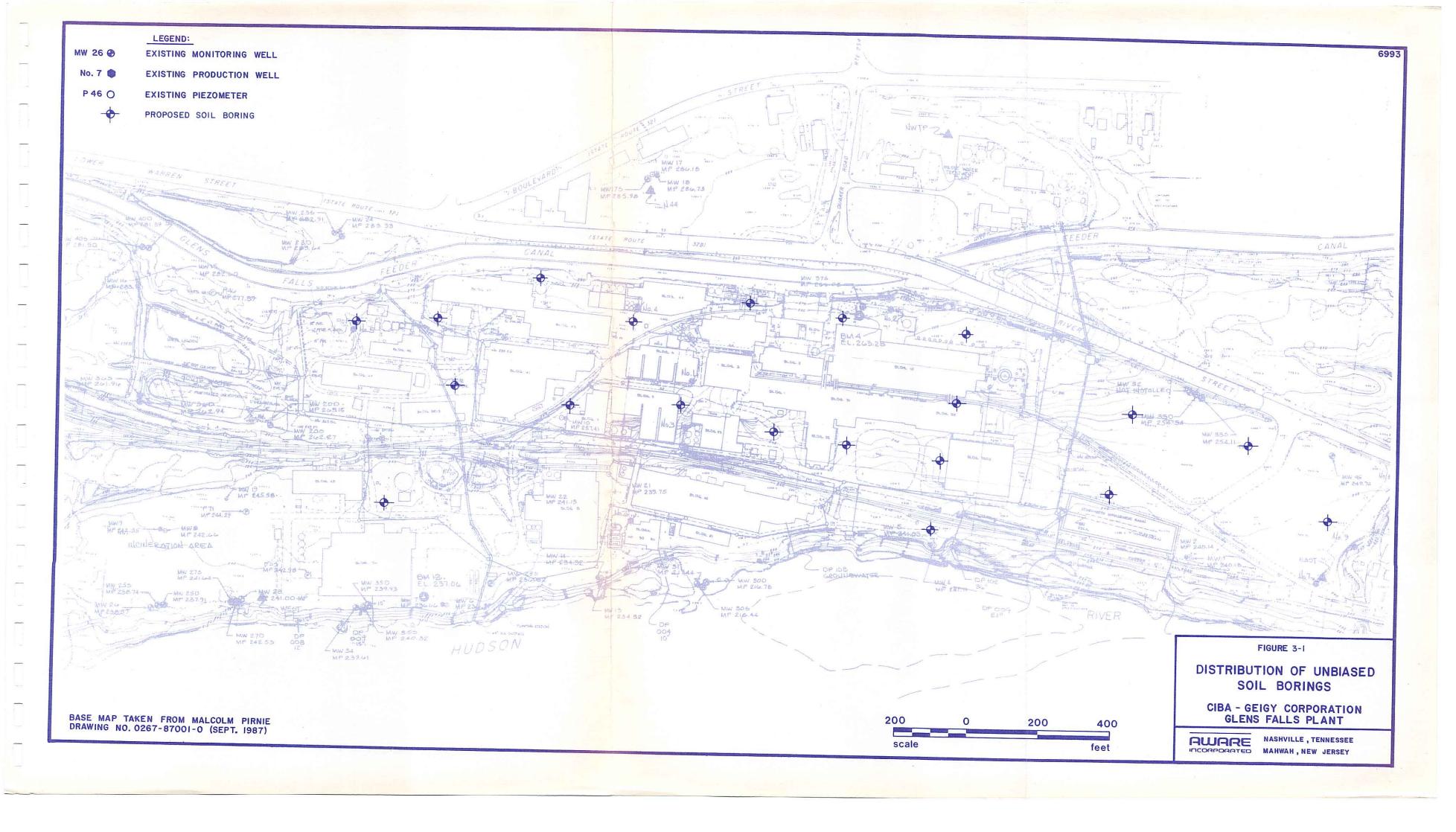
Reconnaissance Borings

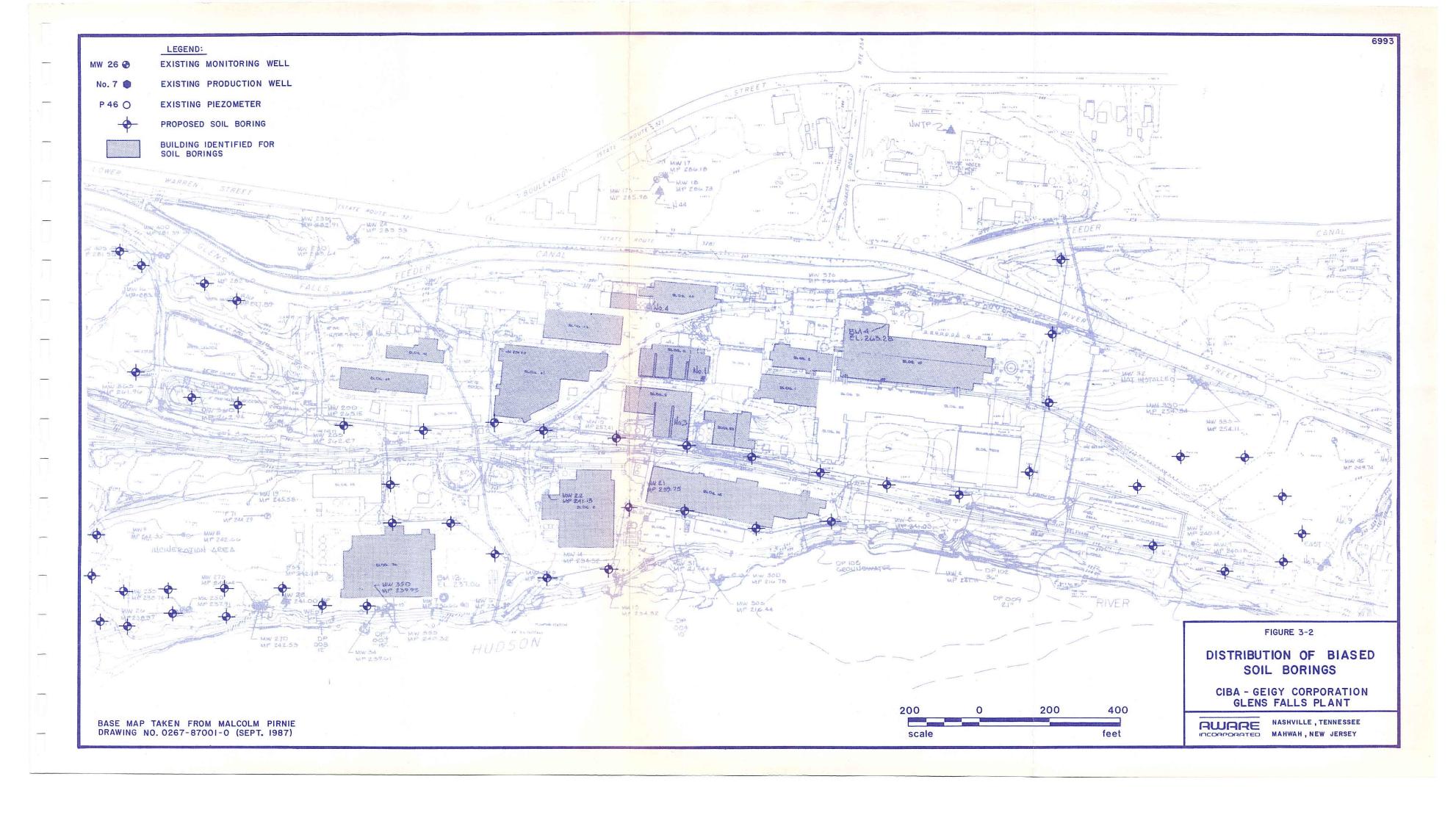
An unbiased grid, consisting of 20 reconnaissance borings, has been developed and is conceptually illustrated on Figure 3-1. Each boring will extend from ground surface to bedrock in accordance with the procedures contained in Appendix A. Furthermore, each soil sample interval (two feet long) will be subjected to both organics (GC) and metals (ICP) screening. Soil samples will be selected for expanded laboratory analysis in accordance with the procedures described in Section 2-2.

North and South Waste Pile Borings

Two borings will be drilled in the north waste pile and two borings in the south waste pile in order to collect samples for the leaching test proposed in Task 4 (Figure 3-2). Previous data have been collected by compositing soils from within four-foot depth increments. This work plan will consist of individual samples, each representing a two-foot long increment. Sample screening will be conducted on each sample as described in Appendix A.

Three off-site borings are also proposed in order to evaluate the extent (if any) of waste pile materials migration toward the west. These borings will, by necessity, require access approval by the current owners of the adjacent property. Exact boring locations will be selected in the field based on





observations of topography and soil color as well as the surface geophysical program (if employed).

Incineration Area Borings

Soil data collected by Malcolm Pirnie indicates the potential for soil contamination in the southwestern portion of the site known as the incineration area. In order to supplement the existing data, nine borings are proposed. Samples will be collected and subjected to metals and organics screening in accordance with the procedures of Appendix A.

Three off-site borings have also been proposed west of the incineration area. The borings will be located based on a visual reconnaissance of the area and the surface geophysical program (if employed). These borings will be contingent upon permission and access limitations.

Parking Lot Borings

Thirteen borings were previously located in the eastern parking lot in the vicinity of what was once a U.S. Government chrome ore stockpile. These borings did not define the limits of this material however, and thus, additional borings are proposed. It is anticipated that eight borings will be sufficient to define this limit. Each boring will provide subsurface samples for visual examination and sample screening.

Building Slab Borings

Upon the complete demolition of selected buildings, one to three borings will be located and drilled through the remaining concrete slab for soil sample collection. The following buildings were selected for this evaluation based on their operational history.

Building	1	Building	42	Building	49
Building	2	Building	43	Building	45
Building	5	Building	10	Building	56
Building	6	Building	44	Building	8
Building	41	Building	53	Building	2128

Prior to exploration, each floor slab will be visually inspected for signs of damage, such as open cracks, missing concrete or other openings which may have served as a passageway for contamination to enter the underlying soil. Care will be taken to ensure that the identified cracks, etc., were present during the operation of the building and are not merely the result of decommissioning activities. The location of any sub-floor structures such as sumps, drains, vaults or tanks will also be identified. Borings will then be located on a case-by-case basis to best evaluate the potential impact on the soil from these areas. Soil sample collection, screening and analysis will be conducted in a similar fashion to the reconnaissance borings.

Industrial Sewer Borings

In order to evaluate the quality of soils associated with the industrial sewer, a program of 24 exploratory borings have been proposed at roughly 200-foot spacings. The borings will be used to collect soil samples for visual inspection and to conduct metals and organics screening. This program will be conducted after the initial boring programs have been implemented.

Task 4 - Laboratory Analysis

Based on the number of proposed soil borings and the anticipated subsurface conditions, approximately 1,000 soil samples will be screened in the laboratory for organics and metals. Of these, approximately 200 will include the "supplemental" metals cyanide and mercury, and approximately five percent or 50 will be selected for the laboratory analysis for the expanded parameter list of metals. The number of samples analyzed for organics will depend upon the results of the GC screening but will be limited to two per boring. All laboratory analyses will be conducted by Aquatec, Inc. of Burlington, Vermont.

Inorganic analysis will be in accordance with New York State protocol and will be analyzed at Level III which includes reporting of internal quality assurance/quality control. For every twenty samples, matrix pike and matrix pike duplicates will be included for quality control.

The expanded list of metals will include the four metals (barium, cadmium, chromium and lead) and will be expanded to include copper, iron, and strontium by ICP. Arsenic and selenium will be analyzed by graphite furnace atomic adsorption, Mercury will be analyzed by cold vapor atomic adsorption. Cyanide will be analyzed by a distillation/colorimetric method.

The parameters to be included for each of these groups are identified in Table 2-1. Approximately 50 percent of the samples selected for the expanded list of metals, will be subjected to the leaching test (approximately 25). This test will be conducted in accordance with the procedures described in Appendix B.

Samples for organics will be analyzed using U.S. EPA OSW Methods 8240 (volatiles) and 8270 (semivolatiles). These analyses will be in accordance with New York State protocol and will be analyzed at Level III which includes reporting of internal quality assurance/quality control. For every twenty samples, matrix spike and matrix spike duplicates will be included for quality control.

Task 5 - Survey

Each of the borings will be surveyed for horizontal location (New York State Plane Coordinates) and elevation (+/- 0.1 feet, USGS Datum). The locations will be marked with wooden stakes by AWARE prior to and after the borings have been completed. The surveyor will be invited to tour the site prior to beginning this work. All survey work will be conducted by a New York State licensed surveyor. The locations will be clearly identified on a site base map along with their respective ground surface elevation.

Task 6 - Data Compilation and Report

All data collected during this investigation, as well as previous soils data, will be placed in a computerized database. Parameters, such as coordinate location, soil type and depth, sample interval, sample screening values and analytical results will be included. Once entered, various data management

procedures will be used to evaluate vertical and/or horizontal relationships (trends). These procedures may include sorting, kriging, graphing or statistical analysis. This data will be presented in both tabular and graphical form.

The data will also be presented on site maps and cross sections. Maps may include the extent and thickness of tailings and natural clay deposits, top of bedrock, top of clay surface and, of course, isoconcentration maps of the various metals and organics of concern. Depending on the results, maps may be prepared for selected depth intervals (i.e., lead concentration at the 2-4 foot depth) or along selected cross sections.

All data, including laboratory QA/QC and the results of the soil screening, would be included. The report would also provide a discussion of the results and recommendations for subsequent action, if necessary.

APPENDIX A Soil Sampling Protocol

APPENDIX A

Soil Sampling Protocol

The collection of soil overburden samples will be required in order to fulfill a variety of objectives including physical description, sample screening, and laboratory chemical analysis. The work plan shall specify the data objective, location, depth, and analytical parameters for each soil sample program. The purpose of this field operation procedure is to describe the methods to be used during each of these activities.

Physical Description

All soil samples will be collected using a two-foot long, standard or three-inch diameter split-spoon sampler. A drilling rig will be used in conjunction with hollow stem augers and the samples will be collected from the ground surface to refusal on bedrock. The samples will be collected in consecutive two-foot long intervals in accordance with ASTM D-1586-87.

For each two-foot interval, the sample will be visually examined and described in accordance with the Unified Soil Classification System. This information, together with a record of the length of the recovered portion of the interval, will be entered into the field log book. A portion of the soil will then be placed in an eight ounce, wide-mouth glass jar using a stainless steel knife. The jar will be filled to three-fourths its volume, and capped with a screw-on lid. The jar lid will then be marked with the following information:

- Date
- Boring Location Number
- Sample Number
- Depth Interval
- Job Number

The sample jar will then be placed in a similarly labeled carton for storage at a designated location.

Sample Screening for Organics

All soil samples collected during this investigation will undergo sample screening as described below.

- A portion of the sample will be immediately placed in a 40 ml sample container upon opening of the split-spoon sampler.
- The container will be immediately capped with a screw-on lid and teflon septum, preserved in accordance with the procedures described below and transported to Aquatec's laboratory.
- The sample will be screened in the lab using the gas chromatography method.

Sample Screening for Metals

All soil samples collected during this investigation will undergo sample screening for metals as described below.

- A portion of the sample will be placed in a 40 ml sample container.
- The sample container will be properly labeled and sent to Aquatec's laboratory in accordance with the procedures described below.
- The sample will then be digested and the extract analyzed for barium, cadmium, chromium and lead by the ICP method.
- Selected samples will also be analyzed for supplemental metals and the expanded list of metals.

Soil Samples for Laboratory Analysis

Soil samples to be collected for laboratory analysis will undergo the identical handling procedures described for visual classification and sample screening. Since the selection of samples for laboratory analysis will be

made only after the sample screening is completed, these samples must be collected for possible analysis from each two foot depth interval. This will require that two additional 40 ml containers be filled for possible organic analysis and one 16 oz. glass container be filled for possible analysis of the expanded list of metals. Samples to be analyzed for organics shall be placed in the containers as quickly as possible. Furthermore, all samples for laboratory analysis shall be preserved and transported in accordance with the following procedures.

All samples to be sent to the laboratory for chemical analysis must be maintained in a condition that is as close as possible to in situ conditions, particularly in the case of the volatile organics. The first consideration is the proper selection of containers, preservation, and associated holding times. Other considerations include proper field notes, proper chain-of-custody procedures, and proper labeling of the samples.

Containers

The type and size of containers used for soil sample collection has been specified in the previous sections and will be provided by Aquatec.

Preservation

The general purpose of preservation is to maintain the original characteristics (and thus validity) of the sample during the time required for shipping of the sample to the laboratory. For soil, the only preservation technique is cooling the sample to approximately 4°C. This will be done in the field using ice or cold packs in coolers.

Sample Custody Procedures

The goal of implementing chain-of-custody procedures is to ensure that the sample is traceable from the time it is collected until it, or its derived data, are used. Samples would be considered "in custody" under the following conditions:

- 1. It is in personal possession
- 2. It is in personal view after being in personal possession
- 3. It was in personal possession when it was properly secured
- 4. It is in a designated secure area

When transferring and/or shipping from the field, samples will be accompanied by the chain-of-custody record (Exhibit A). The form includes the signatures of the relinquishers and the receiver as well as the date and time of the exchange and any pertinent remarks. Since all samples will be immediately placed in coolers, shipment will also be made using these coolers. The samplers will complete the appropriate portion of the chain-of-custody form and hand deliver the cooler to the person transporting the coolers to Aquatec's laboratory. The receiving party will complete the remainder of the form and a copy will be retained by the sampler and kept with the field data sheets for that round of sampling. Each cooler will also be sealed using chain-of-custody tape.

Document Control

A Data Sheet (Exhibit B) is a permanent record of activities relating to the collection of the sample. Information included on the data sheet for this sampling program consist of the names of those responsible for the samples, the date, the location and the reason for the sampling activity. It will also include data on methodology specifically related to the collection of the sample. Finally, the data sheet will contain a detailed description of any apparent characteristics of the sample. Data sheets will be legible, accurate and objective.

Labels

The sample to be sent to the laboratory for chemical analysis will be identified with the following information:

- Date and time of collection
- Boring number

- Sample number
- Analysis to be performed
- Sampler's name and affiliation.

Equipment Cleaning Methods

Equipment in actual contact with a laboratory sample will be cleaned prior to and between each use. The equipment will then be stored in a controlled manner until it is used. Equipment such as split-spoon samplers and soil knives will be cleaned with the following materials:

- Alconox dissolved in clean water
- Clean water rinse
- Nitric acid rinse (not to be used in preparation of samples for cyanide analysis)
- Clean water rinse
- Methanol rinse
- Deionized water rinse
- Air dry.

Non-dedicated drilling and excavation equipment in contact with soil or waste materials will be cleaned prior to use and between each boring location. Decontamination of this equipment will be accomplished using a brush and alconox to remove large solid particles followed by steam cleaning with clean water.



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-		CHAIN OF C	USTODY RECORD)
JOB No. :				
	ENTIFICATI	ON:		
LOCATION NA	LAB SAMPLE No.	co	NTAINERS - NUMBER/TYPE	CONTAINER CONDITIO
CHAIN OF (CUSTODY CH	PONICL E.		
COLLECTED BY		RUNICLE		
NAME:			DATE:	
'				
L	NSFERRED TO:		SEALS PLACED ON CON	TAINERS ? YES D
NAME:			DATE:	TIME:
[2]				
	NSFERRED TO:		ARE SEALS INTACT ?	LI TES LI NO LI N/A
NAMF:			DATE:	TIME:
[3]				☐ YES ☐ NO ☐ N/A
	ABORATORY BY		THE SEASON INTO T	1 120 L NO L NA
NAME:			DATE:	TIME:
DISPOSED BY	`		THE SERES IN FACT.	
5 NAME:				
			HEET" FOR SPECIFIC SAME	PLING DETAILS.
		ANOTHER PARTY ?		



WEST MILFORD, NEW JERSEY NASHVILLE, TENNESSEE

ENVIRONMENTAL SAMPLE FIELD DATA SHEET

7/86

SAMPLE NUMBER

INCORPORATED	SAMPLE NUMBER
PROJECT:	DATE:
CLIENT:	WEATHER CONDITIONS:
JOB No:	AIR TEMPERATURE:
	PERSONNEL:
SAMPLE LOCATION:	LOCATION CODE
SAMPLE SURFACE / MEDIA:	MEDIA CODE
LOCATION COORDINATES:	NORTH EAST 5.
SAMPLE COLLECTION METHOD: 7.	LABORATORY
☐ WOOD SURFACE ☐ WET WI	PE (WETTED WITH HEXANE)
	- GRAB SOIL /SEDIMENT - GRAB
	
SAMPLE TYPE:	
BULK SURFACE X	<u></u>
SAMPLE DESCRIPTION: (SURFACE, MEDIA, MIS	SAMPLE AREA (CM2)
1	
ADDITIONAL SAMPLE DESCRIPTION: 10b.	
COMPOSITE OF LL LOCATIONS	
PENETRATION, MM TO MM QA/QC: REPLICATE OF	M, COLLECTED BELOW (SAMPLE NO.)
FIELD BLANK	PLE NO.)
OTHER DESCRIBE:	
SAMPLING EQUIPMENT: DEL	PREPARED CLEANED DICATED OFF-SITE ON-SITE
EQUIPMENT CLEANING METHOD: WATER WASH SOLVENT WASH SOLVENT TYPE: ACETONE METHANOL	HIGH PURITY SOLVENT RINSE WATER RINSE HEXANE OTHER:
OTHER REMARKS:	

Figure

Appendix B Leaching Test Protocol

Standard Test Method for SHAKE EXTRACTION OF SOLID WASTE WITH WATER¹

This standard is issued under the fixed designation D 3987; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (a) indicates an editional change since the last revision or reapproval.

1. Scope

- 1.1 This test method covers a procedure for leaching of solid waste to obtain an aqueous solution to be used to determine the materials leached under the specified testing conditions.
- 1.2 This test method provides for the shaking of a known weight of waste with water of specified composition and the separation of the aqueous phase for analysis.
- 1.3 This standard may involve hazardous materials, operations, and equipment. This standard does not purpon to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Applicable Documents

- 2.1 ASTM Standards:
- C 471 Method for Chemical Analysis of Gypsum and Gypsum Products²
- D 75 Practice for Sampling Aggregates
- D 420 Practice for Investigating and Sampling Soil and Rock for Engineering Purposes
- D 1129 Definitions of Terms Relating to Water
- D 1193 Specification for Reagent Water
- D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures
- D 2234 Method for Collection of a Gross Sample of Coal³
- D 3370 Practices for Sampling Water
- E 122 Practice for Choice of Sample Size to Estimate the Average Quality of a Lot or Process⁴

3. Definitions

3.1 For definitions of terms used in this test method, see Definitions D 1129.

4. Significance and Use

- 4.1 This test method is intended as a rapid means for obtaining an extract of solid waste. The extract may be used to estimate the release of certain constituents of the solid waste under the laboratory conditions described in this procedure.
- 4.2 This test method is not intended to provide an extract that is representative of the actual leachate produced from a solid waste in the field or to produce extracts to be used as the sole basis of engineering design.
- 4.3 This test method is not intended to simulate site-specific leaching conditions. It has not been demonstrated to simulate actual disposal site leaching conditions.
- 4.4 The intent of this test method is that the final pH of the extract reflect the interaction of the extractant with the buffering capacity of the solid waste.
- 4.5 The intent of this test method is that the water extraction simulate conditions where the solid waste is the dominant factor in determining the pH of the extract.

¹This test method is under the jurisdiction of Committee D-34 on Waste Disposal and is the direct responsibility of Subcommittee D34.02 on Extraction and Leachase Testing.

Current edition approved Oct. 21, 1985. Pablished March 1986. Originally published as D 3987 – 81, Last previous addrson D 1987 – 81.

^{1.} Annual Book of ASTM Standards, Vol 04.01.

Annual Book of ASTM Standards, Vol 04 08.

Annual Book of ASTM Standards, Vol 11.01.
Annual Book of ASTM Standards, Vol 05.05.

Annual Book of ASTM Standards, Vol 14.02



- 4.6 The test method produces an extract that is amenable to the determination of both major and minor constituents. When minor constituents are being determined, it is especially important that precautions are taken in sample storage and handling to avoid possible contamination of the samples.
- 4.7 This test method has been tested to determine its applicability to certain inorganic components in the solid waste. The test method has not been tested for applicability to organic substances and volatile matter (see 5.3).
- 4.8 The agitation technique, rate, and liquid-to-solid ratio specified in the procedure may not be suitable for extracting all types of solid wastes. (See Sections 7, 8, and the discussion in Appendix X1.)

5. Apparatus

- 5.1 Agitation Equipment, of any type that rotates about a central axis at a rate of 29 r/min, Fig. 1. (See discussion of agitation in Appendix X1.)
- 5.2 Membrane Filter Assembly—A borosilicate glass or stainless steel funnel with a flat, fritted base of the same material and membrane filters.
- 5.3 Containers, round, wide-mouth, of a composition suitable to the nature of the solid waste and the analyses to be performed, and constructed of materials that will not allow sorption of constituents of interest. One-gallon (or 4-L) containers should be used with 140-g samples and 1/2-gallon (or 2-L) containers with 70-g samples. Multiples of these sizes may be used for larger samples. The containers should be of the same approximate geometry as the 2-L and 4-L bottles. These sizes were selected to establish suitable geometry and provide that the sample plus liquid would occupy approximately 80 to 90 % of the container. Containers must have a watertight closure. Containers for samples where gases may be released should be provided with a venting mechanism. (Note that the venting of the container has the potential to affect the concentration of volatile extracts in the extract.) Containers should be cleaned in a manner consistent with the analyses to be performed.

6. Reagents

6.1 Purity of Reagents-Reagent grade chemicals shall be used in all tests. Unless otherwise

- indicated, it is intended that all reagents shall conform to the specifications of the American Chemical Society, where such specifications are available. 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.
- 6.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water at 18 to 27°C (Specification D 1193). The method by which the Type IV water is prepared, that is, distillation, ion exchange, reverse osmosis, electrodialysis, should remain constant throughout testing.

7. Sampling

- 7.1 Obtain a representative sample of the solid waste to be tested using ASTM sample methods developed for the specific industry where available. (See Practices D 75 and D 420 and Method D 2234.)
- 7.2 Where no specific methods are available, sampling methodology for materials of similar physical form shall be used.
- 7.3 A minimum sample of 5000 g shall be sent to the laboratory (see Practice E 122).
- 7.4 It is important that the sample of the solid waste be representative with respect to surface area, as variations in surface area would directly affect the leaching characteristics of the sample. Solid waste samples should contain a representative distribution of particles sizes.
- 7.5 Keep samples in closed containers appropriate to the sample type prior to the extraction in order to prevent sample contamination or constituent loss. Where it is desired to extract biologically or chemically active samples in their existing state, store the samples at 4°C (Practices D 3370) and start the extraction within 8 h. Where it is desired to extract such samples in a state representative of the results of biological or chemical activities, the samples may be specifically handled to simulate such activities. Record the storage conditions and handling procedures in the report.

[&]quot;Reagent Chemicals American Chemical Society Specifications." Am. Chemical Soci. Washington, DC, For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards." by Joseph Rosin. D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmatippeta."



g. Sample Preparation

- 8.1 For free-flowing particulate solid wastes. obtain a sample of the approximate size required in the test by quartering the sample (Section 7) received for testing on an impermeable sheet of glazed paper, oil cloth, or other flexible material as follows:
- 8.1.1 Empty the sample container into the center of the sheet.
- 8.1.2 Flatten out the sample gently with a suitable straightedge until it is spread uniformly to a depth at least twice the maximum particle diameter particle size.
- 8.1.3 Remix the sample by lifting a corner of the sheet and drawing it across, low down, to the opposite corner in a manner that the material is made to roll over and over and does not merely dide along. Continue operation with each corner. proceeding in a clockwise direction. Repeat this operation ten times.
- 8.1.4 Lift all four corners of the sheet towards the center and holding all four corners together. raise the entire sheet into the air to form a pocket for the sample.
 - 8.1.5 Repeat 8.1.2.
- 8.1.6 With a straightedge at least as long as the flattened mound of sample (such as a thinedged yard stick), gently divide the sample into quarters. An effort should be made to avoid using pressure on the straightedge sufficient to cause damage to the particles.
 - 8.1.7 Discard alternate quarters.
- 8.1.8 If further reduction of sample size is necessary, repeat 8.1.3 through 8.1.7. A minimum sample size of 70 g is recommended for each extraction. Additional samples should be provided for determination of solids content. If smaller samples are used in the test, report this fact_
- 8.2 For field-cored solid wastes or castings produced in the laboratory, cut a representative section weighing approximately 70 or 140 g for testing, plus samples for determination of solids content. Shape the sample so that the leaching solution will cover the material to be leached.
- 8.3 For fluid solid wastes, mix thoroughly in a manner that does not incorporate air to assure uniformity before withdrawing a 70 or 140 g sample for test. Take samples for determination of solids content at the same time as the test samples.

9. Procedure

- 9.1 Record the physical description of the sample to be tested including particle size so far as it is known.
- 9.2 Solids Content-Determine the solids content of separate portions of the sample as follows:
- 9.2.1 Dry to constant weight two dishes or pans of size suitable to the solid waste being tested at 104 ± 2°C. Cool in a desictator and weigh. Record the value to \pm 0.1 g.
- 9.2.2 Put an appropriately sized portion of sample of the solid waste to be tested into each pan. Scale the weight used to the physical form of the solid waste tested. Use a minimum of 50 g but use larger samples where particles larger than 10-mm in average diameter are being tested. Weigh, Record the weight to ± 0.1 g.
- 9.2.3 Dry 16 to 20 h at 104 ± 2°C. Certain solid wastes, such as scrubber sludges, may contain compounds that are subject to calcination at the specified drying temperature. Dry these compounds at lower temperatures. For example, gypsum may be successfully dried at 45°C (Method C 471) and CaSO₃-1/2H₂O wastes at 85°C. Record the actual temperature and time of the drying period.
- 9.2.4 Cool to room temperature in a desiccator and reweigh. Record the weight to ± 0.1 g.
- 9.3 Shake Procedure—Weigh or tare the container to be used in the shake test to the nearest or within 1 g.
- 9.4 Add to the container approximately 70 g of solid waste (Section 8) and determine and record the weight of sample used to 42 g. If weights other than 70 g are used, note in the ובססוד.
- 9.5 Add to the container a volume of test water (6.2) equal in millilities to 20 times the weight in grams of the sample used in 9.4. See discussion of dilution ratio in Appendix (Exampie: 70 g sample = 1400 mL water).
- 9.6 Agitate continuously for 18 ± 0.25 h at 18 to 27°C.
- 9.7 Open the container. Observe and record any physical changes in the sample and leaching solution.
- 9.8 Let the sample settle for 5 min; then separate the bulk of the aqueous phase from the solid phase by decantation, centrifugation, or filtration through a coarse filter paper as appro-

6

priate. Then vacuum or pressure filter the liquid through a 0.45-um filter. If the separation

µm filter or other device may be used. Record any deviations in the report.

9.9 The filtrate obtained in 9.8 is the extract mentioned elsewhere in this test method. Measure the pH of the extract immediately, then preserve the extract in a manner consistent with the chemical analysis or biological testing procedures to be performed (Practices D 3370). If sufficient liquid phase is not available for the analyses, so indicate in the report and do not continue the procedure; or alternatively, perform the extraction procedure on additional samples of the solid waste to obtain sufficient liquid phase. Where phase separation occurs during the storage of the extract, appropriate mixing should be used to ensure the homogeneity of the extract prior to its use in such analysis or testing.

method results in prolonged filtering time, an 8-

9.10 Analyze the extract for specific constituents or properties or use the extract for biological testing procedures as desired using appropriate ASTM standard methods. Where no appropriate ASTM methods exist, other methods may be used and recorded in the report.

10. Calculation

10.1 Calculate the solids content of the individual samples from the data obtained in 9.2 as follows:

S = A/B

where:

A = weight in grams of sample after drying.

B = original weight in grams of sample, and

S = solid content. g/g

Average the two values obtained. Record as the solids content.

11. Report

11.1 The report shall include the following:

11.1.1 Source of the solid waste, date of sam-

pling, and sample preservation used,

11.1.2 Description of the solid waste including physical characteristics and particle size, if known (9.1),

11.1.3 Solids content (9.2) (see Method D 2216).

11.1.4 Sample weight if other than 70 g.

11.1.5 Drying time and temperature if other than 16 to 20 h at $104 \pm 2^{\circ}C$.

11.1.6 pH and results of specific analyses calculated in appropriate units. State analytical procedures used, and filter used if other than 0.45 µm.

11.1.7 Observation of changes in test material or leaching solution recorded in 9.8.

11.1.8 Date leach testing started, preservation used for extract, and date of analysis.

12. Precision and Bias

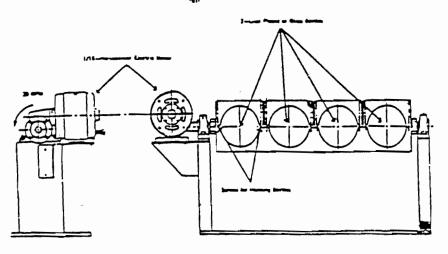
12.1 No information is presently available as to the precision or bias of the analysis of specific constituents in the extract. It is recommended that users of this test method validate the applicability of their chosen methods of detection by spiking portions of the extract, before using these test methods for the analysis of the extract.

12.2 Based on a collaborative series of tests on five solid wastes including fly ash, cutting waste, aluminum industry waste, foundry waste, and non-ferrous foundry waste, the overall precision of this test method was improved. The above data was presented at the second ASTM D-34 Solid Waste Symposium.⁸

12.3 The precision of this test method may vary depending on the solid waste being tested and on the element being extracted.

12.4 Determination of the bias of this test method is not possible, as no standard reference material exists.

⁸ A copy of this paper "Statistical Analysis and Description of Factors Affecting the ASTM Leaching Test" by Dr. Robert Paule can be obtained from ASTM Headquarters.



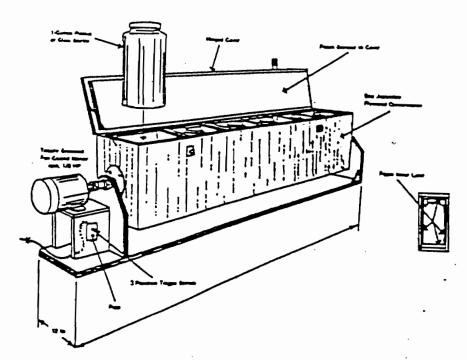


FIG. 1 Extractor

APPENDIX

(Nonmandatory Information)

XI. AGITATION TECHNIQUES AND RATE, AND LIQUID/SOLID RATIOS

X1.1 While a major effort relative to development of the test method has been undestaken to determine the optimum agitation rate, equipment, and liquid/solid ratios specified in the test method, it is recognized that these variables may significantly influence the sults on certain solid wastes, and that they may not be adequate for certain solid wastes, such as monolithic, solidified, or organic wastes.

X1.2 The possible effects of varying the agitation technique and rate include degree of mixing, rate of

release of constituents, and particle abrasion effects. The precision of the test method may also be infla-

X1.3 The possible effects of varying the dilution ratio include degree of mixing, rate of release of constituents (and possible concentration effects, depending on availability), and particle abrasion effects.

X1.4 Efforts are underway to develop test methods that are more suitable for use with organic, monolithic, and solidified wastes.

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