DURACELL INC.
NORTH TARRYTOWN, NEW YORK

QUALITY ASSURANCE PROJECT PLAN FOR CLOSING THE DURACELL INC. FACILITY

PROJECT #425-1
JANUARY 1988

EDER ASSOCIATES
CONSULTING ENGINEERS, P.C.
85 Forest Avenue
Locust Valley, New York 11560

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I. INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents the sampling and analyses which will be performed during closure of the Duracell Inc. facility, located at 60 Elm Street North Tarrytown, New York. Closure consists of the following work tasks:

1. Cleaning of the Plant Building
2. Closure of the Hazardous Waste Storage Facility
3. Demolition of the Plant Building
4. On-Site Soil Remediation
5. Off-Site Soil Remediation

The purposes and scope of the sampling and analysis to be performed under each work task are presented in the following paragraphs:

(1) Cleaning of the Plant Building

Cleaned surfaces of the building structure will be tested to demonstrate that the level of cleanliness described in the report, "Characteristics of Demolition Debris" (Attachment F) has been achieved.

Cleaned equipment will be tested to demonstrate that the remaining surface metal concentrations do not present a health hazard to unprotected workers.

(2) Closure of the Hazardous Waste Storage Facility

Cleaned surfaces of the Hazardous Waste Storage Facility will be tested to document the closure. In addition, the closed Hazardous Waste Storage Facility will be tested in accordance with the rule proposed by the United States Environmental Protection Agency (EPA) for Identification and Listing of Hazardous Waste, 40 CFR 261 as published in the Federal Register Vol. 51, No. 114 FR 21648 June 13, 1986, as required by the Hazardous Waste Facility Closure Plan Approval (refer to Appendix A).

(3) Demolition of the Plant Building

Demolition debris will be tested for parameters required to obtain permission for disposal in a construction and demolition debris landfill. Demolition debris to be used for on-site fill will be tested to demonstrate compliance with soil remediation levels. Materials to be salvaged will be tested to demonstrate that the remaining surface metal concentrations do not present a health hazard to unprotected workers. Air monitoring on off-site properties will be performed during the demolition work to protect public health and environment.

(4) On-Site Soil Remediation

It has been determined that some on-site soils, outside the building envelope, require removal. In addition soils within the building envelope will be sampled and analyzed to determine compliance with remediation levels and assess their condition. The scope of this work task will be modified, if necessary, based on the results of this sampling.

Removed soils will be tested for parameters required to obtain permission for disposal. Sampling and analyses of remaining soil will be conducted after excavation to document that remediation objectives are achieved. Borrow material to be used for backfill will be tested to verify that it is appropriate for possible future uses of the site.

Buried tanks and piping that are to be removed, cleaned and salvaged will be tested to demonstrate that the remaining surface concentrations do not present a health hazard to unprotected workers.

Air monitoring at off-site properties will be performed during soil remediation to protect public health and the environment.

(5) Off-Site Remediation

Removed soils will be tested for parameters required to obtain permission from the industrial waste landfill for disposal. Sampling and analyses of remaining soils will be performed to document the remediation. Borrow material to be used for backfill will be tested to verify that it is appropriate for possible future uses of the properties.

Air monitoring at off-site properties will be performed during soil remediation to protect public health and the environment.

The sampling and analysis to be performed under each work task is presented in Sections III through VII of this report. General procedures for quality assurance and quality control procedures applicable to the sampling and analysis project are presented in Section II. Air monitoring to be performed on off-site properties is presented in Section VIII.

The Contractors will sample and analyze other wastes resulting from their activities in performing the work tasks in accordance with New York State Department of Environmental Conservation (DEC) regulations relating to Hazardous Waste Manifest System and Related Standards for Generators, Transporters and Facilitize (6NYCRR Part 372).

II. GENERAL PROCEDURES

1. Data Quality Requirements and Assessments

Eder Associates Consulting Engineers, P.C. (EA) will be responsible for project coordination and overall Quality Assurance (QA). EA will be responsible for sampling operations and Quality Control (QC) under each work task. Analysis of samples for metal content will be performed by:

ESARCO Inc.
50 South Buckout Street
Irvington, New York 10533

Analysis of samples for other parameters will be performed by:

C.T. Male Associates
50 Century Hill Drive
Latham, New York 12110

The laboratories will be responsible for laboratory QC, data quality review, performance auditing and systems auditing.

2. Sampling Procedures

The following types of samples will be obtained:

surface wipe samples (of the building interior
  and equipment surfaces)
soil samples
core samples (at the Hazardous Waste Storage Facility)
demolition debris

Sampling of demolition debris will be performed using the same general procedures as soil sampling.

2.1 Surface Samples

Building and equipment surfaces to be cleaned will be wipe sampled to determine the residual surface concentrations of lead and mercury. Buried tanks, which may be discovered during on-site soil remediation, will be inspected or sampled to determine the composition of the contents. The surfaces of such tanks will be sampled and analyzed for parameters of concern.

The wipe sampling technique will be used to determine surface concentrations. This technique involves wiping a representative number of areas on the surface to be sampled with an absorbent sampling pad. Each sampling area will be a triangular with six inch orthogonal sides (.125 square feet) and will be thoroughly wiped to remove residues. Pads from all surface areas to be sampled will be placed in one sample container which will be labeled with an indelible pen as follows:

- sample identification number
- number of wipes
- total area wiped
- date
- initials of person collecting the sample

For quality control duplicate and blank samples will be taken for at least 10 percent of the sample matrix. Duplicate samples will be taken by wiping adjacent areas on the surfaces being sampled. Sample containers will be securely closed and placed in shipping overpacks for transportation to the laboratory.

Prior to collecting wipe samples for metal analysis, the sampling pads (kimwipe tissues) will be pre-rinsed in a glass bowl containing distilled water. The bowl will have been washed with an alconox detergent solution and double rinsed with tap water and distilled water prior to sampling each surface. Each sampling area will be wiped with one kimwipe and kimwipes from all areas on the surface being sampled will be placed in one plastic bag. Blanks, consisting of six kimwipes dipped in distilled water, will be prepared during the sampling. The equivalent wipe area is 0.75 square feet.

For determining the surface concentration of organic compounds, the sampling pads will be Whatman No. 4 filter papers. Four filter papers will be used to sample each surface and two of the papers will be randomly placed in each of two sterilized 40 ml glass vials with teflon caps. Each filter paper will be used to wipe either one or two sampling areas depending upon the residues remaining. The equivalent wipe area for each vial will either be 0.25 or 0.50 square feet (SF). Blanks will consist of four filter papers in two vials and the equivalent surface area will be the same as sampling areas.

2.2 Soil Sampling

On-Site and Off-Site soil samples will be taken and analyzed for mercury content, hazardous waste characteristics and other parameters as required by field conditions during performance of the work tasks. Soil samples will be obtained using a stainless steel scoop, 12 inch long steel sampling tube of 3/4 inch diameter or two foot long steel split spoon sampler. Sample locations will be selected to avoid incorporating vegetation in the sample. The sampler will be driven to the required depth and withdrawn carefully to retain the sample undisturbed. Plant materials which are collected with the soil will be carefully removed using clean tweezers prior to transferring the sample into sample containers.

Sampling equipment will be cleaned prior to use at each location and depth by washing with an alconox detergent solution and double rinsing with tap water and distilled water. Samples will be collected and placed in precleaned glass jars with teflon lined lids except those samples to be analyzed for volatile organic compounds (VOCs) which will be placed in two sterilized 40 ml glass vials with teflon lined lids. Sample containers will be labeled as follows:

- sample identification number
- date
- sample depth
- initials of person collecting the sample

The containers will be securely closed and placed in shipping overpacks for transportation to the laboratories.

For quality control duplicate samples will be prepared for at least 10 percent of the sample matrix. Duplicates of samples to be analyzed for parameters other than VOCs will be prepared by placing the sample in a precleaned stainless steel bowl, mixing and splitting into two sample containers. Duplicates of samples to be analyzed for VOCs will be separately collected from the sampling instrument and placed into two glass vials. On each day of soil sampling, a sample of the final rinse distilled water runoff from the sampling instruments will be collected in the same type of containers as the soil samples. Once during each week of soil sampling, a sample of the distilled water used for rinsing the sampling instruments will be collected in the same type of containers as the soil samples.

2.3 Core Samples

Core samples at the Hazardous Waste Storage Facility will be obtained and placed in a wide mouth precleaned glass jar with a teflon lined lid labeled as follows:

- sample identification number
- date
- initials of person collecting the sample

The sample containers will be securely closed and placed in shipping overpacks for transportation to the laboratory where core samples will be pulverized and composited as specified.

Sampling equipment will be washed with an alconox detergent solution and double rinsed with tap water and distilled water.

2.4 Log Book

All information pertinent to the sampling will be recorded in a log book. This will be bound with consecutively numbered pages. As a minimum, entries in the log book will include the following:

- sample identification number
- date and time of sampling
- name of person collecting the sample

For wipe sampling, the surface being sampled and the number and location of wipe samples will be recorded. For soil and core sampling, the location and depth of sample will be recorded. Pertinent observations of field conditions will be recorded to aid in the interpretation of the results.

3. Sample Handling

Samples to be analyzed for metal content will be shipped to ESARCO Inc. on the day of collection. Samples to be analyzed for other parameters will be shipped to C.T. Male Associates by courier on a timely basis to avoid exceeding permissible holding times. Such samples will be stored until shipped at 4°C.

To establish the documentation necessary to trace sample possession from the time of collection, a chain-of-custody record will be filled out and will accompany every sample shipment. A chain-of-custody record is illustrated in Appendix B.

The record will contain the following minimum information:

- sample identification number;
- date and time of sample collection;
- location and method of collection;
- number of containers;
- analysis;
- remarks; and
- signature of persons involved in collection, transportation and reception at the laboratory, and inclusive dates and times of possession.

The samples will be delivered to the person in the laboratory authorized to receive samples who will: inspect the condition of each sample and the sample container seal; reconcile the sample container labeling with that on the chain-of-custody record; assign a laboratory number; log in the sample in the laboratory log book; and store the sample in a secured sample storage room or cabinet until assigned for analysis. Any discrepancies between sample container labeling and the chain-of-custody record will be resolved with EA before the sample is assigned for analysis.

4. Laboratory Analysis

Samples will be analyzed for lead and mercury content, hazardous waste characteristics and for other parameters as required by field conditions. Core samples of the Hazardous Waste Storage

Facility will be tested in accordance with the rule proposed by the EPA on June 13, 1986 for Identification and Listing of Hazardous Waste. The methodology for this test is presented in Appendix A.

The procedures for the analysis of solid samples for lead and mercury content will be in accordance with the latest edition of "Test Methods for Evaluating Solid Wastes", EPA SW-846:

Parameter	Method	Holding Time
Lead	3050: Acid digestion of sediments, sludges & soils 7420: Atomic absorption, direct aspiration	6 months
Mercury	7471: Manual cold vapor technique	28 days

Solid samples will be stored at 4°C and will be extracted as soon as possible and the extract will be analyzed with the allowable holding time.

The total metal content of all kimwipes in a sample bag will be determined and will be divided by the corresponding total wipe area to determine the areal metal concentration on the surface which has been sampled.

Soil samples will be analyzed for total solids in accordance with Method 160.3 of the latest edition of, "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020. Metal content of soils will be reported on a dry weight basis.

The procedures for the analysis of liquid samples for lead and mercury content will be in accordance with the latest edition of "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020:

Parameter	Method	Holding Time
Lead	239.1: Atomic absorption, direct aspiration	6 months
Mercury	2451: Manual cold vapor technique	28 days

Upon arrival at the laboratory, liquid samples will be preserved by the addition of nitric acid to a pH<2 and will be stored at 4°C until analysis within the allowable holding time.

Analysis for hazardous waste characteristics will be in accordance with EPA Regulations for Identifying Hazardous Waste 40 CFR 261.

Other analysis, which may be required as a result of field conditions, will be performed in accordance with the previously referenced documents and the latest edition of "Test Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater" EPA-600/4-82-057.

5. Laboratory Quality Control

Quality control includes: calibration procedures and preventive maintenance data reduction, validation and reporting performance and system audits corrective action.

5.1 Calibration Procedures and Preventive Maintenance

Each laboratory will routinely calibrate and maintain its equipment in order to insure the validity of the analytical results. The equipment manufacturer will recommend the frequency at which the equipment should undergo routine maintenance checks. No analyses will be conducted unless the equipment satisfies the criteria established by the manufacturer. All calibrations and equipment checks will be documented by the individual conducting the test.

Laboratory equipment will be calibrated prior to analysis for each parameter. Calibration curves will be composed of a minimum of three standards and one reagent blank. The procedures outlined in the manufacturer's specifications and "Test Methods For Evaluating Solid Waste", EPA SW-846, November 1986 will be strictly followed in order to insure the validity of the results.

5.2 Data Reduction, Validation and Reporting

The laboratories will develop quality control charts as outlined in EPA's "Handbook of Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, March 1979, to establish comparable precision and accuracy limits. If comparable limits cannot be achieved, the laboratory will review their findings with the Engineer, adjust the limits as agreed, and take any necessary corrective action before continuing the analyses.

Quality control protocols, to be used to insure that the data quality requirements are being met, will include a minimum of at least one laboratory duplicate and one spike analysis for each parameter. Additional quality control checks will include analyzing a reagent blank and midpoint standard for each parameter. New standard curves will be established with each new batch of reagents, using at least seven concentration levels. A comprehensive outline of quality control requirements for analyses of the metals by atomic absorption is presented in Appendix C. The "optional requirements" listed will be considered mandatory.

Data will be considered valid when analyses yield results within quality control limits, as defined by the quality control charts and quality assurance objectives for precision and accuracy. These analyses include: external performance evaluation audits; split sample analyses; duplicate sample analyses; spiked sample analyses; detection limits analyses; and instrument calibrations.

The laboratories will document all the quality control criteria that will be used to reduce and validate data. This includes the number of data used to develop control charts, and specific precision and accuracy data for each parameter being analyzed.

The laboratories will issue reports to the Engineer summarizing the analytical results and quality assurance/quality control data. This information will be incorporated in the documentation report which will include assessment of the results from performance and system audits, internal quality control and quality assurance problems, including their remedies. The documentation report will include a separate quality assurance section summarizing all information provided by the laboratories.

5.3 Performance and System Audits

In order to monitor the capability and performance of the total measurement system, it will be necessary to conduct performance and system audits.

The system audit will determine if all field and laboratory procedures are being conducted in accordance with the QA Program. During the project, EA will supervise all field work to verify that the project plan and Standard Operating Procedures are being implemented. It will be the responsibility of the certified laboratory to conduct its own system audit on its operational systems and physical facilities.

Performance audits will be conducted by analysis of aqueous and solid samples for selected parameters sent to the laboratory.

5.4 Corrective Action

Corrective actions will be initiated if any of the following events occur:

(1) Failure to calibrate field equipment;

(2) Failure to calibrate laboratory equipment; and

(3) Failure of data to fall within QC limits, as defined by the laboratory's quality control charts.

The corrective action procedure for each listed item is presented below:

(1) Repair or replace field equipment. Check calibration.

(2) Follow procedures outlined by manufacturer.

(3) Check for sample interferences. Check calibration.

The individual or laboratory conducting the test will be responsible for initiating the corrective action. All problems and solutions will be documented and all analyses since the last in-control point must be repeated or discarded.

III. SAMPLING DURING BUILDING CLEANING

1. Scope of Work

The plant building is shown in Drawing No. 1. The building is one story, except for the northwest portion, which is two stories. The north central portion has an attic space with loose insulation. Below grade construction includes a boiler room at the center of the west wall of the building.

The building, having 58 different rooms or functional areas in which a variety of manufacturing operations and administrative functions had been performed, has been divided into 15 areas which are designated in the drawing. The areas are defined by use and isolated by enclosure walls and are generally served by separate air handling systems. Area 15, outside the southeast corner of the building, is a concrete block storage shed of three rooms and an exterior concrete pad of about 600 square feet. Two of the rooms and the pad are the Hazardous Waste Storage Facility.

The building is concrete slab on grade construction, with brick, cinder block and concrete block exterior walls. The boiler room and the second floor are poured concrete. Roofs, except above Area 10, are wood with either shingle or PVC membrane covering. The roof above Area 10 is metal decking and composition roofing with gravel. Some interior rooms have internal roofs. The Drawing shows some of the air conditioning and exhaust systems, floor trenches, manholes and drains in the building. The drawing also shows air pollution control equipment and chemical storage tanks located in the areaway at the southeast corner of the building. Major mechanical equipment remaining in the building includes a freight elevator.

The scope of the building cleaning work includes:

- removal of ductwork
- cleaning of air pollution control equipment, storage tanks, other mechanical equipment and roof
- cleaning interior building surfaces

Operations for cleaning building surfaces are presented in Table 1. Special operations include removal of duct and piping containing asbestos, cleaning the elevator shaft, and removal of attic insulation. The scope of the work does not include Area 4, which had been previously cleaned, or outside Shed Room 59.

The wipe sampling technique will be used to determine the surface concentration of lead and mercury remaining on building surfaces and equipment after cleaning. Performance specifications for the cleaning work are:

Cleaned Building Surfaces:

Average residual surface concentration any one metal: not to exceed 0.1 mg/SF

Cleaned Equipment

Residual surface concentrations: lead not to exceed 16.6 mg/SF; mercury not to exceed 0.34 mg/SF.

Sampling of Area 15, which includes the Hazardous Waste Storage Facility, is discussed in the next section.

2. Building Surface Sampling

Building surfaces which had been cleaned and will be sampled include floors, surfaces in Areas 9 and 14 and room 58 which were pressure washed, and floor trenches, pits and manholes.

Surface sampling is summarized in Table 2. Drawing No. 2 shows

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TABLE 1

CLEANING OPERATIONS

	Operation	Area 1	Area 2	Area 3	Area 5	Area 6	Area 7		Area 8
							Rm 28	Rm 40/46A	
1	Clean AHU's	X	X	X			X	X	
2.	Clean Trenches, Manholes and Pits			X			X		
3.	Vacuum Floors	X	X	X	X	X	X	X	X
4.	Pressure Wash Surfaces								
5.	Scrub Floors	X	X	X	X	X	X	X	X
6.	Special Operations								

Table 1 Continued . . .

Operation	Area 9	Area 10	Area 11	Area 12	Area 13	Area 14	Area 15	Rm 16	Rm 34	Rm 58
1. Clean AHU's				X						
2. Clean Trenches, Manholes and Pits		X						X		
3. Vacuum Floors	X	X	X	X	X	X	P	X	X	X
4. Pressure Wash Surfaces	X					X	P			X
5. Scrub Floors		X	X	X			P	X	X	
6. Special Operations	X	X			X					
Comments				1.			2.	3.		4.

AHU: Air Handling Unit
P : Partial
Comment 1.: Includes stairwells to the first floor and boiler room levels.
Comment 2.: Vacuum and power wash all interior surfaces; scrub concrete pad.
Comment 3.: Do not scrub platform 16A.
Comment 4.: Wash storage tank.

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TABLE 2

BUILDING SURFACE SAMPLING

Location	Floor Area (SF)	Surface	Number of Wipes	Sample Identification
Area 1	2,500	Floors	6	1B
Area 2	2,800	Floors	6	2B
Area 3	16,000	Floors	10	3B
Area 5	620	Floors	4,4	4B, 28B
Area 6	1,360	Floors	4	5B
Area 7	1,950	Floors	6	6B
Area 8	3,300	Floors	6	7B
Area 9	1,660	Floors	6	8B
		Walls	9	9B
		Ceilings	6	10B
Area 10	14,000	Floors	10,10	11B, 29B
Area 11	4,700	Floors	6	12B
Area 12	9,280	Floors	8	13B
Area 13	8,000	Floors	8	14B
Area 14	980	Floors	6	15B, 30B
		Walls	6	16B
		Ceilings	6	17B
Room 58	110	Floors	4	18B
		Walls	6	19B
		Ceilings	4	20B
Room 34	190	Floors	4	21B

Table 2 Continued . . .

Location	Floor Area (SF)	Surface	Number of Wipes	Sample Identification
East Floor Trench	--	Floors	9	22B
West Floor Trench	--	Floors	9	23B
Pits and Manholes	--	Floors/Walls	4 each	24B through 27B
Blanks	--	--	6 each	31B through 33B

building conditions after cleaning and the location of each wipe sample on floors, walls and ceilings. Wall wipe samples will be taken approximately equidistant between the floor and ceiling. Ceiling wipe samples will be taken directly above floor wipe samples. The drawing does not show the location of wipe samples to be taken in floor trenches, pits and manholes.

Wipe samples in floor trenches will be taken at nine equally spaced locations along the centerline of each trench floor. The plan includes an allowance for sampling four manholes and pits. Other pits and manholes, uncovered during the cleaning work, will be similarly sampled. One wipe sample will be taken at the center of each pit and manhole floor, if possible, and three wipe samples will be taken on walls halfway between the floor and top. If a floor wipe is not possible, then four wall wipe samples will be taken. In rectangular pits and manholes, one wall wipe sample will be taken along the vertical centerline of each wall. In circular pits, equidistant wall wipe samples will be taken along the horizontal circumference line.

Twenty-seven different surfaces will be sampled, including the allowance for four manholes and pits. Quality control samples will include three duplicate samples, 28B, 29B and 30B, and three blank samples, B31B, 32B and 33B.

3. Equipment Wipe Sampling

Equipment surfaces which had been cleaned and will be sampled include the interior and exterior surfaces of air pollution control equipment and fans, the exterior surfaces of boiler room equipment and the interior surfaces of air handling units. Surface sampling is summarized in Table 3.

All mechanical equipment located in the areaway at the southeast corner of the plant building is to be cleaned. The equipment

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TABLE 3

EQUIPMENT SURFACE SAMPLING

Location	Surface	Number of Wipes	Sample Identification
Cyclones	Interior	6	1E, 8E
	Exterior	6	2E
Baghouses	Interior	9	3E
	Exterior	9	4E
Area 14 (Boiler Room)	Exterior	10	5E
Fans*	Exterior/Interior	6	6E
Air Handling Units*	Interior	6	7E
Blanks		6	9E

NOTE: * - three randomly selected units

includes air pollution control equipment consisting of two cyclones, three baghouses and appurtenances. Both the interior and exterior surfaces of the cyclones and baghouses will be sampled. The samples should be representative of the surface metal concentrations remaining on all other equipment in the areaway. One sample will be obtained from the interior surfaces of the cyclones by wipe sampling at three different accessible locations in each cyclone. One sample will be obtained from the exterior surfaces of the cyclones by wipe sampling at three randomly selected locations on each cyclone. The baghouses will be similarly sampled.

All exposed surfaces in the boiler room (Area 14) will be pressure washed. One sample of the exterior surfaces of the boiler room equipment will be obtained by wipe sampling ten randomly selected locations.

Other equipment to be cleaned includes all surfaces of fans and the interior air passage surfaces of air handling units (AHUs). Three randomly selected fans and AHUs should be representative of all fans and AHUs and will be sampled. One wipe sample of the fans will be obtained by wipe sampling at one randomly selected interior location and at one randomly selected exterior location on each fan. One wipe sample of the AHUs will be obtained by wipe sampling at two randomly selected locations within the air passages of each AHU.

Seven different surfaces will be sampled. Quality control samples will include one duplicate sample, 8E, and one blank sample, 9E.

IV. SAMPLING DURING CLOSURE OF THE HAZARDOUS WASTE STORAGE FACILITY AND ROOM 52

1. Scope of Work

Area 15, outside the southeast corner of the building, is a concrete block storage shed of three rooms and an exterior concrete pad of about 600 square feet. Two of the rooms and the pad are the Hazardous Waste Storage Facility. The facility will be closed in accordance with the Closure Plan of October, 1985 and Addendum Report No. 1 of April, 1986 approved by the DEC on January 20, 1987. The area will be cleaned as part of the building cleaning task as specified in Table 1. Dirt and grit on the interior floors of the rooms will be removed by vacuuming and all interior surfaces of the rooms will be pressure washed and the concrete pad will be scrubbed.

The wipe sampling technique will be used to determine the surface concentration of lead and mercury remaining on the surfaces of the Hazardous Waste Storage Facility and other room after cleaning. Performance specifications for the cleaning work are that the average residual surface concentration of any metal shall not exceed 0.1 mg/SF. In addition, core sampling of the Hazardous Waste Storage Facility Structure will be performed for testing in accordance with the proposed EPA rule for identification of hazardous waste.

2. Surface Sampling

Surface sampling is summarized in Table 4. Drawing No. 2 shows the location of each wipe sample on floors, walls and ceilings.

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TABLE 4

AREA 15 SURFACE SAMPLING

Surface	No. of Wipes	Sample Identification
HAZARDOUS WASTE STORAGE FACILITY		
Floors	6	34B, 40B
Walls	6	35B
Ceiling	4	36B
ROOM 52		
Floors	4	37B
Walls	4	38B
Ceiling	4	39B
BLANKS	6	41B

Separate samples will be taken in the Hazardous Waste Storage Facility and room 52. Wall wipe samples will be taken approximately equidistant between the floor and ceiling. Ceiling wipe samples will be taken directly above interior floor wipe samples. Quality control samples will include one duplicate sample, 40B, and one blank sample, 41B.

3. Core Sampling

Core samples of the Hazardous Waste Storage Facility will be taken as follows:

Location	Number of Core Samples
floors	3
walls	4
ceilings	2

Floor samples will be taken at the center of each of the two rooms and the concrete pad. Wall samples will be taken on opposite walls of the two rooms approximately equidistant between the floor and ceiling. Ceiling samples will be taken directly above interior floor samples. The samples, designated C-1 through C-9, will be delivered to the laboratory where they will be combined on an equal weight basis into one composite sample for analysis.

V. SAMPLING DURING BUILDING DEMOLITION

1. Scope of Work

Demolition includes removal of structures above grade level and floor slabs except the Hazardous Waste Storage Facility floor slab. Inert debris will be used to fill below grade construction and about 700 cubic yards will be stockpiled on-site. Demolition debris to be used for fill or to be stockpiled will be sampled and tested to verify that its mercury concentration is below remediation levels. Other demolition debris will be transported and disposed to a permitted construction and demolition (C&D) landfill.

Steelwork, mechanical and electrical equipment will be removed, cleaned as required, and salvaged. The wipe sampling technique will be used to verify the effectiveness of cleaning. Performance specifications for cleaning are that residual surface concentrations of lead shall not exceed 16.6 mg/SF and mercury shall not exceed 0.34 mg/SF.

During the demolition, floor slabs of below grade construction will be broken up. These are shown on Drawing No. 3 and include the boiler room, elevator shaft, East and West Floor trenches, interior pits and manholes, exterior pits and truck bay. Prior to breaking these floor slabs, the below grade construction will be inspected to determine their condition and to locate cracks where leakage into underslab soils might have occurred. Following the breaking of the floor slabs, underslab soils will be inspected to identify visually stained areas.

A plan for sampling soils beneath these floor slabs is proposed in this section which will be modified in accordance with the inspections. The soils will be sampled, analyzed for mercury content and screened for the presence of VOCs using an Organic Vapor Analyzer (OVA). Those samples indicating the presence of VOCs or which are visually stained will also be analyzed for the presence of organic compounds. If the soil mercury concentration is below the remediation levels and soil conditions are determined to be satisfactory, the below grade construction will be filled with acceptable inert demolition debris.

2. Salvage Wipe Sampling

Steelwork and equipment to be salvaged will be cleaned and placed in a roll-off bin. When the contractor proposes to remove the bin from the site, he will notify Duracell's "Resident Project Representative" (RPR) and the bin contents will be sampled. The bin will be divided into quadrants and one wipe sample will be taken at the center of each quadrant. All wipe samples from each bin will be combined into a single composite sample for lead and mercury analysis. For quality control, duplicate and blank samples will be taken for at least 10 percent of the sample matrix.

3. Soil Sampling

The proposed sampling of soils is summarized in Table 5 and sampling locations are shown on Drawing No. 3. At the time of construction, imported fill, which had not been exposed to battery manufacturing operations, may have been placed as a foundation material and exposed on-site soils may be one or two feet below the construction. At each sample location, soil samples will be obtained in six (6) inch increments to a depth of at least three feet and designated by the suffix letter A through F, respectively.

Each soil sample will be divided into three portions. One set of portions from each location will be screened for the presence of VOCs, the second will be retained for laboratory analysis, if required, and the third will be retained for mercury analysis.

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TABLE 5

SOIL SAMPLING
BELOW GRADE CONSTRUCTION

Below Grade Construction	No. of Sample Locations	Sample Identification(1)
Boiler Room	2 North	S-1
	2 South	S-2
Elevator Pit	2	S-3
Truck Bay	4	S-4, S-19
East Floor Trench	6	S-5 through S-10
West Floor Trench	1	S-11, S-20
Exterior Pits (3)	1 each	S-12 through S-14
Interior Pits & Manholes (4)	1 each	S-15 through S-18

NOTE: (1) Suffix letter indicates depth of sample as follows:

Suffix Letter	Depth, in.
A	0-6
B	6-12
C	12-18
D	18-24
E	24-30
F	30-36

Stained soil samples will be inspected with respect to color, consistency and odor to determine whether the stain is due to the presence of inorganic metals, petroleum products or other organic compounds. If the 30-36 inch deep sample indicates the presence of VOCs or is visually stained, deeper samples will be taken in the same manner until VOC-free or visually clean soil is encountered. At locations where VOCs are present as determined by the OVA or the soil is stained, the portion of the second sample set corresponding to the sample depth of highest measured VOCs or most intense stain will be sent to the laboratory to be analyzed for VOCs, metals petroleum hydrocarbons or organic compounds if their presence is suspected. The first sample set and those portions of the second sample set not sent to the laboratory will be returned to the sample location site.

Equal volumes of the portions of the third sample set taken at the same depth will be combined as indicated in Table 5 to prepare composite samples S-1, S-2, S-3, S-4 and S-19. Alternate depth samples of the third sample set will be analyzed for mercury content. As a minimum the following samples will be analyzed for mercury content:

Depth in.	Suffix Letter
0-6	A
12-18	C
24-30	E

Deeper samples will also be analyzed if these have been taken. If the mercury concentration of any of the samples exceeds the remediation levels, then the next deeper sample will be analyzed to define the depth of penetration. If the mercury concentration of the deepest sample exceeds the remediation values, additional soil sampling will be performed during on-site soil remediation as presented in the following section.

The plan provides for soil sampling beneath twelve below grade structures. The boiler room has a north part in Building 1 and a south part in Building 6. The soil beneath the floor slab of each part has a different history of exposure and separate composite soil samples will be prepared from each part for analysis of mercury content. Sample series S-1 (A through F, etc.) will be a composite of samples taken at two equidistant locations along the centerline of the north part, and sample series S-2 will be a composite of samples taken at two equidistant locations along the centerline of the south part. In the elevator pit, sample series S-3 will be a composite of samples taken at two equidistant locations along the centerline. The truck bay will be divided into four quadrants, and sample series S-4 will be a composite of samples taken at the center of each quadrant. Sample series S-5 through S-10 will be taken along the centerline of the East Floor Trench and each series will be analyzed separately. The West Floor Trench is a pipe trench to which wastewater was not normally discharged. Extensive investigation of soils beneath this trench is not required unless inspection shows deteriorated construction or stained soils. Sample series S-11 will be taken at the centerline of the trench in Building 5. Sample series S-12 through S-18 will be taken at the center of each interior and exterior manhole and pit and each series will be analyzed separately.

Quality control samples will include obtaining 12 duplicate samples, series S-19 and S-20, which will be analyzed in the ratio of one each per ten samples analyzed.

Additional samples will be taken for analysis if required by conditions encountered. Other interior manholes and pits uncovered during the demolition work will be similarly sampled and additional quality control samples will be taken as required.

4. Sampling of Demolition Debris

An estimated 500 cubic yards of debris can be used to fill below grade construction and 700 cubic yards will be stockpiled on-site to fill other on-site excavations. This debris will be sampled as placed. One sample will be obtained from each 300 cubic yards, to be designated D-1 through D-4. Each sample of debris will be approximately one pound in weight with a maximum particle size of 1/2 inch. Quality control samples will include one duplicate sample, D-5 taken separately and not split. The samples will be analyzed for mercury content.

VI. SAMPLING DURING ON-SITE SOIL REMEDIATION

1. Scope of Work

This work task includes excavation of on-site soils having mercury concentrations exceeding remediation levels or which are otherwise unsatisfactory and removal of concrete slabs, paving, buried tanks and plumbing drain piping. Excavated soils, concrete, paving, drain piping and wastes resulting from the work will be transported to permitted disposal facilities. Removed tanks and connected piping will be cleaned and salvaged. Demolition debris stockpiled on-site and borrow materials will be used to backfill excavated areas to grade. Prior to the start of this work task, the contractor will furnish a representative sample of borrow material from each source. The samples will be analyzed for mercury content and screened for the presence of VOCs using the OVA. If the measured concentrations are within background concentrations, the source will be considered acceptable.

The known extent of soils to be excavated and the fuel oil tank and drain piping to be removed are shown on Drawing No. 3. After demolition of the building and prior to any on-site remediation, a survey of the site and additional soil sampling will be performed to identify the extent of any other soils required to be excavated and other removals to be performed.

The survey will:

(a) identify presently unknown buried tanks and their contents;

(b) verify the location and condition of plumbing drain lines;

and

(c) delineate any areas of visually stained soils.

The additional soil sampling plan will be modified to include sampling below grade construction not backfilled during demolition and areas of visually stained soils which may be discovered during the survey. The scope of soil excavation, tank removal and drain piping removal, will be modified in accordance with the findings of the site survey and soil sampling.

Soils identified as having mercury concentrations exceeding remediation levels or which are otherwise unsatisfactory and are to be excavated will be sampled and analyzed to determine the appropriate disposal facilities. Soil remaining after excavation will be sampled and analyzed to verify that remediation levels have been achieved and that remaining conditions are satisfactory. The proposed plan of post excavation soil sampling is based on the known extent of soils to be excavated and will be modified to include other areas where soils are to be excavated.

Soils required to be excavated for the removal of tanks and drain piping will be stockpiled. The stockpiled and remaining soil where tanks and drain piping were removed will be inspected and visually stained soil will be sampled and analyzed. Acceptable soil will be backfilled and unacceptable soil will be removed to appropriate disposal facilities.

The wipe sampling technique will be used to verify the effectiveness of cleaning tanks and connected piping to be salvaged. Performance specifications as appropriate for cleaning are that residual exterior surface concentrations of lead shall not exceed 16.6 mg/SF and mercury shall not exceed 0.34 mg/SF. Interior surfaces of the tanks shall be cleaned of residues.

2. Site Survey

The site survey will include visual inspection and the use of magnetic locators to identify presently unknown buried tanks and verify the location of plumbing drain lines. The condition of exposed drain lines will be inspected to identify locations of potential leakage. Areas of visually stained soils will be delineated during the inspection.

Buried tanks identified during the survey will be inspected to determine the composition of the contents. If materials remain in such tanks, a representative sample will be obtained using a Coliwasa tube and the composition will be determined by inspection or analysis for USEPA priority pollutants with an EPA/NIH/NBS library search of non-priority pollutant organics.

3. Additional Soil Sampling

The proposed additional sampling of soils is summarized in Table 6 and sampling locations are shown on Drawing No. 3. At each sample location, soil samples will be obtained in six (6) inch increments to a depth of at least three feet, designated by the suffix letter A through F, respectively.

This plan of additional soil sampling will be modified to include sampling areas of visually stained soils identified during the survey. Soil sampling will be performed at the center and along the perimeter of each stained area at intervals of about 30 feet and at a minimum of four locations.

Each soil sample will be divided into three portions. One set of portions from each location will be screened for the presence of VOCs, the second will be retained for laboratory analysis, if required, and the third will be retained for mercury analysis. Stained soil samples will be inspected with respect to color, consistency and odor to determine whether the stain is due to the presence of inorganic metals, petroleum products or other organic compounds. If the 30-36 inch deep sample indicates the presence of VOCs or is visually stained, deeper samples will be taken in

DURACELL INC.
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TABLE 6

SOIL SAMPLING WITHIN BUILDING ENVELOPE

Building	Number Of Sample Locations	Sample Identification(1)
1	5	S-101 through S-105, S-125
2	-	----
3	4	S-106
4	-	----
5	9	S-107 through S-115, S-126
6	8	S-116 through S-124, S-127

NOTE: (1) Suffix letter indicates depth of sample as follows:

Suffix Letter	Depth, in.
A	0-6
B	6-12
C	12-18
D	18-24
E	24-30
F	30-36

the same manner until VOC-free or visually clean soil is encountered. At locations where VOCs are present as determined by the OVA or the soil is stained, the portion of the second sample set corresponding to the sample depth of highest measured VOCs or most intense stain will be sent to the laboratory to be analyzed for VOCs, metals, petroleum hydrocarbons or organic compounds if their presence is suspected. The first sample set and those portions of the second sample set not sent to the laboratory will be returned to the sample location site.

Equal volumes of the portions of the third sample set taken at the same depth will be combined as indicated in Table 6 to prepare composite sample series S-106. Alternate depth samples of the third sample set will be analyzed for mercury content. As a minimum the following samples will be analyzed for mercury content:

Depth in.	Suffix Letter
0-6	A
12-18	C
24-30	E

Deeper samples will also be analyzed if these have been taken. If the mercury concentration of any of the samples exceeds the remediation levels, then the next deeper sample will be analyzed to define the depth of penetration. If the mercury concentration of the deepest sample exceeds the remediation values, additional deeper soil sampling will be performed.

A site history and the results of prior underslab soil sampling performed in August 1986 are presented in Appendix D. Buildings 1, 2 and 3 existed when Duracell purchased the property. Prior underslab soil samples taken beneath Buildings 1 showed mercury concentrations below remediation levels except at Location C-11 at the north end of Building 1. Additional soil mercury concentration data within the south end of the building envelope will become available as a result of soil sampling to be performed during building demolition. Sample series S-101 through S-105 is designed to define the extent of elevated mercury concentrations at the north end of the building. Prior underslab soil samples taken beneath Building 2 showed mercury concentrations below remediation values. Additional soil mercury concentration will become available along the periphery of the building as a result of soil sampling to be performed during building demolition and sampling to be performed within the envelope of Building 5. No further soil sampling within the envelope of Building 2 is required. Soils within the envelope of Building 3 will not have been investigated and composite sample series S-106 will be taken. The area within the envelope of this building will be divided into four quadrants and soil samples will be taken at the center of each quadrant. Equal volumes of samples taken at the same depth will be combined for analysis.

Building 4 was the first building constructed after Duracell acquired the property. Prior underslab soil samples taken beneath the building showed soil mercury concentrations below remediation levels. Additional soil mercury concentration data along the East Floor Trench will become available as a result of soil sampling to be performed during building demolition. No further sampling within the envelope of this building is required unless inspection shows the presence of stained soils.

Building 5, constructed at a later date, contains a plumbing drain line along its entire length and several pits. A prior underslab soil sample taken beneath the building showed soil mercury concentration above the remediation levels. Additional soil mercury data will become available in the West Floor Trench, pits and manhole as a result of soil sampling to be performed during building demolition. Sample series S-107 through S-115 are designed to define the extent of soils having mercury concentrations exceeding remediation values.

Building 6 is about 14,000 square feet in area. Prior underslab soil samples were taken at two locations beneath the building. At both locations, soil mercury concentrations increased with depth and exceeded remediation levels 12 to 18 inches below the floor slab at location C-7, at the south end of the building. Additional soil mercury data will become available at the east corners of the building as a result of soil sampling to be performed during building demolition. Sample series S-116, S-117, and S-118 will be taken in the vicinity of Location C-7 to define the extent of soils having mercury concentrations exceeding remediation values. Sample series S-119 through S-124 will be taken throughout the building to define soil conditions within those areas of the building not previously investigated.

Quality control samples will include obtaining 18 duplicate samples, series S-125, S-126 and S-127, which will be analyzed in the ratio of one each per ten samples analyzed.

4. Sampling and Analysis for Soil Disposal

The known extent of soils to be excavated, shown on Drawing No. 3, is divided into four areas. Area 1 is a cone shaped volume of soils. Area 2 is the remaining volume of soils at the southeast corner of the site, including the known buried fuel oil tank, which is to be excavated to a depth of 10 feet below grade. Areas 3 and 4 are exposed soils between paving and the buildings.

The proposed sampling is summarized in Table 7 and sampling locations are shown on Drawing No. 3. At each location, a continuous soil sample will be obtained to the depth indicated on the Table. Samples taken at locations within each area will be combined on a volume basis in proportion to depth. The combined sample will be well mixed and a 500 gram composite sample will be withdrawn and analyzed for hazardous waste characteristics. Quality control samples will include a duplicate sample, S-205.

DURACELL INC.
NORTH TARRYTOWN, NEW YORK

TABLE 7

ON-SITE SOIL SAMPLING FOR DISPOSAL

Remediation Area	Sample Location	Depth Below Grade (ft)
1	S-201, S-205	5
2	S-202-1	7.5
	S-202-2	10
	S-202-3	5
	S-202-4	5
3	S-203-1	6
	S-203-2	6
4	S-204	3.5

Additional sampling using a similar method will be performed in any other areas where soils require excavation and off-site disposal.

5. Post Excavation Soil Sampling

After excavation of soils, the remaining soils will be sampled and analyzed to document that remediation levels have been achieved. Each excavated area will be divided into horizontal segments of about 200 square feet each. Samples will be taken to a depth of 0 to 6 inches at the center of each segment. At least one sample will be obtained from each excavated area. Samples shall also be taken from the walls of excavations extending greater than 5 feet below grade. Each wall of such excavations shall be divided into vertical segments of about 100 square feet each. Samples will be taken from 0 to 6 inches long at the center of each segment. At least one sample will be obtained from each wall. For Quality control duplicate samples will be taken for at least 10 percent of the sample matrix.

Each soil sample will be screened for the presence of VOCs and will be analyzed by the laboratory for mercury content and VOCs if their presence is indicated by the OVA. If remediation levels have not been achieved, or unsatisfactory conditions remain, additional excavation will be performed and the sampling and analysis of remaining soils will be repeated.

6. Tank and Drain Pipe Bedding Soil Sampling

Soils required to be excavated for the removal of tanks and plumbing drain piping will be stockpiled on and covered with polyethylene sheeting. Tanks and drain piping will be visually inspected to determine whether leakage might have occurred. Stockpiled and remaining soils will be inspected for evidence of leakage or staining and sampling and analysis will be performed where evidence of such conditions are observed.

Stockpiles will be sampled at a central location and a continuous sample through the entire depth of the stockpile will be withdrawn. Visually stained soil areas remaining at the location of removed tanks and drain piping will be sampled in accordance with the protocols described in paragraph 3 of this section.

Each soil sample will be divided into two portions. One set of portions from each location will be screened for the presence of VOCs, and the second will be retained for laboratory analysis. In areas, where VOCs are detected, or the soil is stained, the portion of the second sample set, taken within the area, corresponding to the highest measured VOCs or most intense stain will be sent to the laboratory to be analyzed for VOCs, metals, petroleum hydrocarbons or organic compounds if their presence is suspected. The first sample set and those portions of the second sample set not sent to the laboratory will be returned to the sample location site.

If the analysis demonstrates that the stockpile or remaining soils are unacceptable, additional sampling will be performed following the same methodology to determine the extent of unacceptable soils and modify the scope of soil excavation to be performed. Samples of such soil to be excavated will be obtained and analyzed to determine the appropriate disposal facility as described in paragraph 4 of this section.

7. Wipe Sampling of Salvage

The surfaces of tanks and piping which have been cleaned will be sampled. One wipe sample will be taken from every 200 square feet of tank area. A minimum of four wipe samples will be taken from each tank and composited for analysis. A minimum of six wipe samples will be taken on random exterior areas of cleaned piping and composited for analysis. For quality control duplicate and blank samples will be taken for at least 10 percent of the sample matrix. Wipe samples shall be analyzed for parameters of concern identified by inspection.

VII. SAMPLING DURING OFF-SITE SOIL REMEDIATION

1. Scope of Work

This work task includes excavation of soils to a depth of six (6) inches on properties adjacent to the Duracell property as shown in Drawing No. 4. Excavated soils will be transported to permitted disposal facilities. Borrow materials will be used to backfill excavations. Prior to the start of this work task, the contractor will furnish a representative sample of borrow material from each source. The samples will be analyzed for mercury content. If the measured concentration is within background concentration, the source will be considered acceptable.

Soil to be excavated will be sampled and analyzed to determine the appropriate disposal facilities. Soil remaining after excavation will be sampled and analyzed to document the mercury concentration.

2. Sampling and Analysis of Soil to be Disposed

Soil to be excavated will be sampled and analyzed for hazardous waste characteristics. Sampling locations are shown on Drawing No. 4. Two composite soil samples will be obtained. One composite sample, to be designated S-301, will consist of eight samples taken on properties along Elm Street and Kendall Avenue. A second composite sample, to be designated S-302, will consist of seven samples taken on the 30 Andrews Lane property.

One sample will be taken for approximately every 1200 square feet of area to be excavated. At each location, soil samples will be obtained from 0 to 6 inches wide below grade. Samples will be composited on a equal volume basis. For quality control, a duplicate composite sample, to be designated S-303, will be obtained on the 30 Andrews Lane property.

3. Post Excavation Soil Sampling

After excavation of soils, the remaining soils will be sampled and analyzed to document their mercury concentration. Sampling locations are shown on Drawing No. 4. Two composite soil samples will be obtained. One composite sample, to be designated S-304 will be obtained from the 52 Elm Street property and one composite sample, to be designated S-305 will be obtained from the 31 Kendall Avenue property. The excavated area of each property will be divided into four quadrants. Samples will be taken 0-6 inches below the excavated grade at the center of each quadrant. The samples from each property will be composited on an equal volume basis for analysis. Quality control samples will include one duplicate sample, to be designated S-306.

VIII. AIR SAMPLING

1. Protection of Ambient Air Quality

The Contract Documents for Building Demolition, On-Site Soil Remediation and Off-Site Soil Remediation require the Contractor to perform the work tasks using such procedures and methods as are required to protect air quality at off-site properties. The principal potential fugitive emissions of concern are lead, mercury and particulates. Ambient air quality standards for the pertinent parameters as specified by the EPA and DEC are presented in Table 8. Duracell will conduct air monitoring on off-site properties during the performance of the work tasks and will notify the Contractor when violations of ambient air quality standards are likely to occur. Air quality measurements shall be compared to the lowest standard presented in Table 8. The Contractor is required to immediately take such corrective actions as are necessary to protect ambient air quality including but not limited to increased dust suppression measures, constructing temporary barrier walls or cessation of activities until meterological conditions change.

This section proposes a plan of air sampling to be performed during building demolition, on-site and off-site soil remediation. The plan will be modified to include other contaminants which may be identified during the Work Tasks. Following a review of the initial results obtained during each work task, the plan may be modified with respect to parameters and frequency of measurement.

2. Procedures

Control of fugitive emissions shall be performed using direct reading instruments to determine ceiling concentrations as follows:

DURACELL INC.
NORTH TARRYTOWN, NEW YORK

TABLE 8

AMBIENT AIR QUALITY STANDARDS

	24 Hr Avg	30 Day Avg	60 Day Avg	90 Day Avg	Annual Avg
Lead, ug/m^3					
EPA				1.5	
Particulate Matter, ug/m^3					
EPA	150				50
DEC Level II	250	100	85	80	55
Settleable Particulates, mg/cm^2/mo					
DEC					0.30-0.45
Mercury (in organic), ug/m^3					
DEC	0.33 Acceptable Ambient Air Level				

Note: EPA - Federal Environmental Protection Agency (40 CFR 50)
DEC - New York Department of Environmental Conservation
(6 NYCRR PART 257 and Air Guide - 1)

Lead - detector tube
mercury - gold film mercury vapor analyzer
particulates - light scatter aerosol monitor

The presence of organic vapors will also be measured at the same time as ceiling concentrations are measured using an OVA with a detection limit of 1 ppm.

Documentation of ambient air quality will be performed by measuring time weighted average (TWA) concentrations of lead, mercury and particulates using the procedures specified in the latest edition of the NIOSH Manual of Analytical Methods. The procedures are presented in Appendix E.

2.1 Meterological Station

A portable meterological station will be maintained on the Duracell parking lot on the west side of Andrews Lane to continuously measure and record wind speed and direction during building demolition, on-site soil remediation and off-site soil remediation.

2.2 Background Ambient Air Quality

Prior to the start of the work tasks, existing ambient air quality will be measured on the Duracell parking lot. On each of three days, ceiling concentrations will be measured once every two hours for eight hours and 8-hour TWA measurements will be made.

2.3 Air Sampling During Building Demolition and On-Site Soil Remediation

Air quality measurements will be made at seven locations around the Duracell property shown on Drawing No. 4. Ceiling measurements will be made once every two hours and 8-hour TWA samples will be obtained at each location while work tasks are being performed. One or more of TWA samples will be analyzed to confirm that air quality standards are not exceeded. TWA samples to be analyzed will be selected at downwind locations of the Duracell property and at locations where the highest ceiling concentrations are measured.

2.4 Air Sampling During Off-Site Remediation

Air quality measurements will be made at two locations on properties adjacent to or at a maximum distance of 50 feet from the area being remediated. Ceiling measurements will be made once every two hours and 8-hour TWA measurements will be made each working day.

3. Quality Assurance and Quality Control

Quality Assurance and Quality Control Procedures will be similiar to those presented in Section II including maintenance of log book, sample handling and laboratory quality control procedures.

Air monitoring shall be documented by the following information:

- Site Location/Date
- Work Process/Operation Name
- Analysis Method Used
- Air Flow Calibration Record
- Temperature at Sample Location
- Area/Sampling Location Diagram
- Area Sample Description/Location
- Sampling Data
  - Pump I.D.
  - Flow Rate
  - Sample Filter/Tube Number
  - Pump On/Off (time)
  - Lab Sample Number
  - Analysis Results

APPENDIX A

HAZARDOUS WASTE FACILITY CLOSURE PLAN APPROVAL

New York State Department of Environmental Conservation
202 Mamaroneck Avenue, White Plains, New York 10601

Henry G. Williams
Commissioner

RECEIVED
AT EA
JAN 22 1987
FILE NO. 4051
LJE____ FHI____
SJO____ GAR ✓
JH ✓ WJC____
OTHER

January 20, 1987

Mr. Clifford Perman
Duracell Inc.
Berkshire Industrial Park
Bethel, Connecticut 06801

Re: Duracell Inc.
Hazardous Waste Facility
ID #000692913
Closure Plan Approval

Dear Mr. Perman:

This letter is to inform you that, upon review of our records, it is deemed that the applicable requirements for acceptable closure of the above referenced facility have been met, and hereby, approval of the Closure Plan submitted in October 1985 and Addendum Report No. 1 dated April 1986 is granted. This approval is granted based on the following special condition: samples taken at the site during closure activities shall be analyzed for the Proposed Toxicity Characteristic Contaminants and Regulatory Levels published in the June 13, 1986 Federal Register.

Any further investigation and any necessary remediation of the Duracell site will be addressed in a site remediation plan which will be subject to a separate regulatory activity at a later date.

Please note that this approval in no way precludes Duracell's responsibility to submit a closure certification as noted in the Closure Plan. It is deemed that closure of the above referenced facility is not complete, and hence, your firm remains subject to all applicable regulatory fees, until such time that the engineering certification is received by this office.

The aforementioned certification should be forwarded within 30 days from the date of closure to:

Robert J. Haggerty, Jr.
Acting Supervisor
Permit Section, Division of Solid and Hazardous Waste
New York State Department of Environmental Conservation
50 Wolf Road, Room 401
Albany, New York 12233

James Reidy, P.E.
Regional Hazardous Waste Engineer
New York State Department of Environmental Conservation
202 Mamaroneck Avenue, Room 304
White Plains, New York 10601

Richard A. Baker
Chief Permit Administrative Branch
U.S. Environmental Protection Agency
26 Federal Plaza
New York, New York 10278

If you should have any questions or comments regarding the above, please contact Ms. Mariana Dominguez, of my staff, at (914) 761-6660.

Very truly yours,

James Reidy, P.E.
Regional Hazardous Waste Engineer
Region 3

JR:MD:bz
cc: Paul Counterman
Philip Zigarelli, Mayor, Village of North Tarrytown
Robert Ponzini, Esq., Village Attorney
Gary Rozmus, Eder Associates
Michelle M. Taylor

Wastes. Volume 1. EPA Contract 68-03-2552. January, 1981.

(20) National Research Council (NRC). Drinking Water and Health. Vol. 4. Safe Drinking Water Committee. National Academy Press. Washington, D.C. 1982.

(21) Office of Management and Budget (OMB). Interim Regulatory Impact Analysis Guidance. Washington D.C. June, 1981.

(22) Research Triangle Institute (RTI). Regulatory Impact Analysis for Expansion of Toxicity Characteristic Under RCRA. U.S. EPA Contract 68-01-7075. October, 1985.

(23) S-Cubed. Precision Evaluation of the TCLP Protocol For Non-Volatile Components. Draft Report. U.S. EPA Contract 68-03-1958. January 1986.

(24) Spellenberg, S.P. Organic Extraction Procedure. U.S. EPA Contract 68-01-6149. January, 1982.

(25) Technology Applications Inc. (TAI). Statistical Analysis of TCLP Development Data. U.S. EPA Contract 68-01-6936. May 28, 1985.

(26) U.S. EPA. Background Document, Section 261.24, Characteristic of Extraction Procedure Toxicity. National Technical Information Service (NTIS) PB 81-185-027. Springfield, Virginia, May, 1980.

(27) U.S. EPA. Test Methods for Evaluating Solid Wastes—Physical/Chemical Methods, Second ed. Government Printing Office (GPO) 055-002-81001-2. EPA SW-846. Washington, D.C. 1982.

(28) U.S. EPA. Guidelines for Performing Regulatory Impact Analysis. Washington, D.C. December, 1983.

(29) U.S. EPA Science Advisory Board (SAB). Report on the Review of EP-III. Washington, D.C. May, 1984.

(30) U.S. EPA. Background Document: Issues Relating to the Development and Use of Reference Doses to Support 40 CFR Part 268, Land Disposal Restrictions. Washington, D.C. November, 1985.

(31) U.S. EPA. Acceptable Daily Intake Workgroup Paper: Assessing Risks Associated With Systemic Toxicants. Washington, D.C. 1985.

(32) U.S. EPA. Verified Reference Doses (RfD's) of the U.S. EPA. Washington, D.C. 1985.

(33) U.S. EPA. Background Document For Toxicity Characteristic Leaching Procedure. Washington, D.C. February, 1986.

List of Subjects in 40 CFR Parts 261, 271, and 302

Administrative practice and procedure, Air pollution control, Chemicals, Confidential business information, Hazardous materials, Hazardous materials transportation, Hazardous substances, Hazardous waste, Indian lands, Intergovernmental relations, Natural resources, Nuclear materials, Penalties, Pesticides and pests, Radioactive materials, Recycling, Reporting and recordkeeping requirements, Superfund, Water pollution control, Water supply, Waste treatment and disposal.

Dated: May 31, 1986.

Lee M. Thomas,

Administrator.

For the reasons set out in the preamble, it is proposed to amend Title 40 of the Code of Federal Regulations as follows:

PART 261—IDENTIFICATION AND LISTING OF HAZARDOUS WASTE

1. The authority citation for Part 261 continues to read as follows:

Authority: Secs. 1006, 2002(a), 3001, and 3002 of the Solid Waste Disposal Act, as amended by the Resource Conservation and Recovery Act of 1976, as amended (42 U.S.C. 6905, 6912(a), 6921, and 6922).

2. § 261.24 is revised to read as follows:

§ 261.24 Toxicity characteristic.

(a) A solid waste exhibits the characteristic of toxicity if, using the test methods described in Appendix II or equivalent methods approved by the Administrator under the procedures set forth in §§ 260.20 and 260.21, the extract from a representative sample of the waste contains any of the contaminants listed in Table 1 at the concentration equal to or greater than the respective value given in that Table. Where the waste contains less than 0.5 percent filterable solids, the waste itself, after filtering using the methodology outlined in Appendix II, is considered to be the extract for the purpose of this section.

(b) A solid waste that exhibits the characteristic of toxicity, but is not listed as a hazardous waste in Subpart D, has the EPA Hazardous Waste Number specified in Table 1 which corresponds to the toxic contaminant causing it to be hazardous.

TABLE 1.—TOXICITY CHARACTERISTIC CONTAMINANTS AND REGULATORY LEVELS

HWNO and contaminant	CASNO	Regulatory level (mg/1)
D018—Acrylonitrile	107-13-1	5.0
D004—Arsenic	7440-38-2	5.0
D005—Barium	7440-39-3	100
D019—Benzene	71-43-2	0.07
D020—Bis(2-chloroethyl) ether	111-44-4	0.05
D006—Cadmium	7440-43-9	1.0
D021—Carbon disulfide	75-15-0	14.4
D022—Carbon tetrachloride	56-23-5	0.07
D023—Chlordane	57-74-9	0.03
D024—Chlorobenzene	108-90-7	1.4
D025—Chloroform	67-66-3	0.07
D007—Chromium	1333-82-0	5.0
D026—o-Cresol [1]	95-48-7	10.0
D027—m-Cresol [1]	108-39-4	10.0
D028—p-Cresol [1]	106-44-5	10.0
D016—2,4-D	94-75-7	1.4
D029—1,2-Dichlorobenzene	95-50-1	4.3
D030—1,4-Dichlorobenzene	106-46-7	10.8
D031—1,2-Dichloroethane	107-06-2	0.40
D032—1,1-Dichloroethylene	75-35-4	0.1
D033—2,4-Dinitrotoluene	121-14-2	0.13
D012—Endrin	72-20-8	0.003
D034—Heptachlor (and its hydroxide)	76-44-8	0.001
D035—Hexachlorobenzene	118-74-1	0.13
D036—Hexachlorobutadiene	87-68-3	0.72
D037—Hexachloroethane	67-72-1	4.3
D038—Isobutanol	78-83-1	36
D008—Lead	7439-92-1	5.0
D013—Lindane	58-89-9	0.06
D009—Mercury	7439-97-6	0.2
D014—Methoxychlor	72-43-5	1.4
D039—Methylene chloride	75-09-2	8.6
D040—Methyl ethyl ketone	78-93-3	7.2
D041—Nitrobenzene	98-95-3	0.13
D042—Pentachlorophenol	87-86-5	3.6
D043—Phenol	108-95-2	14.4
D044—Pyridine	110-86-1	5.0
D010—Selenium	7782-49-2	1.0
D011—Silver	7440-22-4	5.0
D045—1,1,1,2-Tetrachloroethane	630-20-6	10.0
D046—1,1,2,2-Tetrachloroethane	79-34-5	1.3
D047—Tetrachloroethylene	127-18-4	0.1
D048—2,3,4,6-Tetrachlorophenol	58-90-2	1.5
D049—Toluene	108-88-3	14.4
D015—Toxaphene	8001-35-2	0.07
D050—1,1,1-Trichloroethane	71-55-6	30
D051—1,1,2-Trichloroethane	79-00-5	1.2
D052—Trichloroethylene	79-01-6	0.07
D053—2,4,5-Trichlorophenol	95-95-4	5.8
D054—2,4,6-Trichlorophenol	88-06-2	0.30
D017—2,4,5-TP (Silvex)	93-76-5	0.14
D055—Vinyl chloride	75-01-4	0.05

[1] o-, m-, and p-Cresol concentrations are added together and compared to a threshold of 10.0 mg/1.

3. Appendix II of Part 261 is revised to read as follows:

Appendix II—Toxicity Characteristic Leaching Procedure (TCLP)

1.0 *Scope and application.*

1.1 The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual contaminants are not present in the waste, or that they are present, but at such low concentrations that the appropriate regulatory thresholds could not possibly be exceeded, the TCLP need not be run.

2.0 *Summary of method* (See Figure 1).

2.1 For wastes containing less than 0.5% solids, the waste, after filtration through a 0.6–0.8 μm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than 0.5% solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced (if necessary), weighed, and extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatiles (See Table 1). Following extraction, the liquid extract is separated from the solid phase by 0.6–0.8 μm glass fiber filter filtration.

2.3 If compatible (e.g. precipitate or multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract and these liquids are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield volume weighted average concentration.

3.0 *Interferences.*

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 *Apparatus and materials.*

4.1 Agitation Apparatus: An acceptable agitation apparatus is one which is capable of rotating the extraction vessel in an end-over-end fashion (See Figure 2) at 30±2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction vessel:

4.2.1 Zero-Headspace Extraction Vessel (ZHE). When the waste is a being tested for mobility of any volatile contaminants (See Table 1), an extraction vessel which allows for liquid/solid separation within the device, and which effectively precludes headspace (as depicted in Figure 3), is used. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without having to open the vessel (See Section 4.3.1). These vessels shall have an internal volume of 500 to 600 ml and be equipped to accommodate a 90 mm filter. Suitable ZHE devices known to EPA are identified in Table 3. These devices contain viton O-rings which should be replaced frequently.

4.2.2 When the waste is being evaluated for other than volatile contaminants, an extraction vessel which does not preclude headspace (e.g., 2-liter bottle) is used. Suitable extraction vessels include bottles made from various materials, depending on the contaminants to be analyzed and the nature of the waste (See Section 4.3.3). These bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Section 4.3.2 is used for initial liquid-solid separation and final extract filtration.

4.3 Filtration devices:

4.3.1 Zero-Headspace Extractor Vessel (See Figure 3): When the waste is being evaluated for volatiles, the zero-headspace extraction vessel is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter, and be able to withstand the pressure needed to accomplish separation (50 psi).

Note. When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the extract.

4.3.2 Filter Holder. When the waste is being evaluated for other than volatile compounds, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation is used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressure up to 50 psi and more. The type of filter holder used depends on the properties of the material to be filtered (See Section 4.3.3). These devices shall have a minimum internal volume of 300 ml and be equipped to accommodate a minimum filter size of 47 mm. Filter holders known to EPA to be suitable for use are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene, or polyvinyl chloride may be used when evaluating the mobility of metals.

4.4 Filters: Filters shall be made of borosilicate glass fiber, contain no binder materials, and have an effective pore size of 0.6–0.8 um, or equivalent. Filters known to EPA to meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid washed prior to use by rinsing with 1.0 N nitric acid followed by three consecutive rinses with deionized distilled water (minimum of 500 ml per rinse). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: Any of the commonly available pH meters are acceptable.

4.6 ZHE extract collection devices: TEDLAR* bags or glass, stainless steel or PTFE gas tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device.

4.7 ZHE extraction fluid collection devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a constant displacement pump, a gas tight syringe, pressure filtration unit (See Section 4.3.2), or another ZHE device).

4.8 Laboratory balance: Any laboratory balance accurate to within ±0.01 grams may be used (all weight measurements are to be within ±0.1 grams).

5.0 *Reagents.*

5.1 Water: ASTM Type 1 deionized, carbon treated, decarbonized, filtered water (or equivalent water that is treated to remove volatile components) shall be used when evaluating wastes for volatile contaminants. Otherwise, ASTM Type 2 deionized distilled water (or equivalent) is used. These waters should be monitored periodically for impurities.

5.2 1.0 N Hydrochloric acid (HCl) made from ACS Reagent grade.

5.3 1.0 N Nitric acid (HNO_3) made from ACS Reagent grade.

5.4 1.0 N Sodium hydroxide (NaOH) made from ACS Reagent grade.

5.5 Glacial acetic acid (HOAc) made from ACS Reagent grade.

5.6 Extraction fluid:

5.6.1 Extraction fluid #1: This fluid is made by adding 5.7 ml glacial HOAc to 500 ml of the appropriate water (See Section 5.1), adding 64.3 ml of 1.0 N NaOH, and diluting to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

5.6.2 Extraction fluid #2: This fluid is made by diluting 5.7 ml glacial HOAc with ASTM Type 2 water (See Section 5.1) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

Note.—These extraction fluids shall be made up fresh daily. The pH should be checked prior to use to insure that they are made up accurately, and these fluids should be monitored frequently for impurities.

5.7 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 *Sample Collection, preservation, and handling.*

6.1 All samples shall be collected using a sampling plan that addresses the consideration discussed in "Test Methods for Evaluating Solid Wastes" (SW–846).

6.2 Preservatives shall not be added to samples.

6.3 Samples can be refrigerated unless it results in irreversible physical changes to the waste.

6.4 When the waste is to be evaluated for volatile contaminants, care must be taken to insure that these are not lost. Samples shall be taken and stored in a manner which prevents the loss of volatile contaminants. If possible, any necessary particle size reduction should be conducted as the sample is being taken (See Step 8.5). Refer to SW–846 for additional sampling and storage requirements when volatiles are contaminants of concern.

6.5 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. If they need to be stored, even for a short period of time, storage shall be at 4°C and samples for volatiles analysis shall not be allowed to come into contact with the atmosphere (i.e., no headspace).

7.0 *Procedure when volatiles are not involved.*

Although a minimum sample size of 100 grams is required, a larger sample size may be necessary, depending on the percent solids of the waste sample. Enough waste sample should be collected such that at least 75 grams of the solid phase of the waste (as determined using glass fiber filter filtration), is extracted. This will insure that there is adequate extract for the required analyses (e.g., semivolatiles, metals, pesticides and herbicides).

The determination of which extraction fluid to use (See Step 7.12) may also be conducted at the start of this procedure. This determination shall be on the solid phase of the waste (as obtained using glass fiber filter filtration).

7.1 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative subsample of the waste (100 gram minimum) and proceed to Step 7.11.

7.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device discussed in Section 4.3.2, and is outlined in Steps 7.3 to 7.9.

7.3 Pre-weigh the filter and the container which will receive the filtrate.

7.4 Assemble filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (See Section 4.4).

7.5 Weigh out a representative subsample of the waste (100 gram minimum) and record weight.

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*TEDLAR is a registered trademark of DuPont.

7.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.7 Transfer the waste sample to the filter holder.

Note.—If waste material has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.5, to determine the weight of the waste sample which will be filtered.

Gradually apply vacuum or gentle pressure of 1–10 psi until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., does not result in any additional filtrate within any 2 minute period), filtration is stopped.

Note.—Instantaneous application of high pressure can degrade the glass fiber filter, and may cause premature plugging.

7.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

Note.—Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid—but even after applying vacuum or pressure filtration, as outlined in Step 7.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the extraction as a solid.

7.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (See Step 7.3) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (See Step 7.15) or stored at 4°C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Step 7.5 or 7.7. Record the weight of the liquid and solid phases.

Note.—If the weight of the solid phase of the waste is less than 75 grams, review Step 7.0.

7.10 The sample will be handled differently from this point, depending on whether it contains more or less than 0.5% solids. If the sample obviously has greater than 0.5% solids go to Step 7.11. If it appears that the solid may comprise less than 0.5% of the total waste, the percent solids will be determined as follows:

7.10.1 Remove the solid phase and filter from the filtration apparatus.

7.10.2 Dry the filter and solid phase at 100±20°C until two successive weighings yield the same value. Record final weight.

7.10.3 Calculate the percent solids as follows:

Weight of dry waste and filters minus tared weight of filters divided by initial weight of waste (Step 7.5 or 7.7) multiplied by 100 equals percent solids.

7.10.4 If the solid comprises less than 0.5% of the waste, the solid is discarded and the liquid phase is defined as the TCLP extract. Proceed to Step 7.14.

7.10.5 If the solid is greater than or equal to 0.5% of the waste, return to Step 7.1, and begin the procedure with a new sample of waste. Do not extract the solid that has been dried.

Note.—This step is only used to determine whether the solid must be extracted, or whether it may be discarded unextracted. It is not used in calculating the amount of extraction fluid to use in extracting the waste, nor is the dried solid derived from this step subjected to extraction. A new sample will have to be prepared for extraction.

7.11 If the sample has more than 0.5% solids, it is now evaluated for particle size. If the solid material has a surface area per gram of material equal to or greater than 3.1 cm², or is capable of passing through a 9.5 mm (0.375 inch) standard sieve, proceed to Step 7.12. If the surface area is smaller or the particle size is larger than that described above, the solid material is prepared for extraction by crushing, cutting, or grinding the solid material to a surface area or particle size as described above. When surface area or particle size has been appropriately altered, proceed to Step 7.12.

7.12 This step describes the determination of the appropriate extracting fluid to use (See Sections 5.0 and 7.0).

7.12.1 Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer a 5.0 gram portion to a 500 ml beaker or erlenmeyer flask.

7.12.2 Add 96.5 ml distilled deionized water (ASTM Type 2), cover with watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is < 5.0, extraction fluid #1 is used. Proceed to Step 7.13.

7.12.3 If the pH from Step 7.12.2 is >5.0, add 3.5 ml 1.0 N HC1, slurry for 30 seconds, cover with a watchglass, heat to 50°C, and hold for 10 minutes.

7.12.4 Let the solution cool to room temperature and record pH. If pH is <5.0, use extraction fluid #1. If the pH is >5.0, extraction fluid #2 is used.

7.13 Calculate the weight of the remaining solid material by subtracting the weight of the sub-sample taken for Step 7.12, from the original amount of solid material, as obtained from Step 7.1 or 7.9. Transfer remaining solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

Note.—If any of the solid phase remains adhered to the walls of the filter holder, or the container used to transfer the waste, its weight shall be determined, subtracted from the weight of the solid phase of the waste, as determined above, and this weight is used in calculating the amount of extraction fluid to add into the extractor bottle.

Slowly add an amount of the appropriate extraction fluid (See Step 7.12), into the extractor bottle equal to 20 times the weight of the solid phase that has been placed into the extractor bottle. Close extractor bottle tightly, secure in rotary extractor device and rotate at 30 ± 2 rpm for 18 hours. The temperature shall be maintained at 22 ± 3 °C during the extraction period.

Note.—As agitation continues, pressure may build up within the extractor bottle (due to the evolution of gasses such as carbon dioxide). To relieve these pressures, the extractor bottle may be periodically opened and vented into a hood.

7.14 Following the 18 hour extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter as outlined in Step 7.7. This new filter shall be acid washed (See Section 4.4) if evaluating the mobility of metals.

7.15 The TCLP extract is now prepared as follows:

7.15.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 7.14 is defined as the TCLP extract. Proceed to Step 7.16.

7.15.2 If compatible (e.g., will not form precipitate or multiple phases), the filtered liquid resulting from Step 7.14 is combined with the initial liquid phase of the waste as obtained in Step 7.9. This combined liquid is defined as the TCLP extract. Proceed to Step 7.16.

7.15.3 If the initial liquid phase of the waste, as obtained from Step 7.9, is not or may not be compatible with the filtered liquid resulting from Step 7.14, these liquids are not combined. These liquids are collectively defined as the TCLP extract, are analyzed separately, and the results are combined mathematically. Proceed to Step 7.16.

7.16 The TCLP extract will be prepared and analyzed according to the appropriate SW-846 analytical methods identified in Appendix III of 40 CFR 261. TCLP extracts to be analyzed for metals shall be acid digested. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to 0.1 ml), conduct the appropriate analyses, and combine the results mathematically by using a simple weighted average:

$$\text{Final contaminant concentration} = \frac{(V_1)\,(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (l)
C_1 = The concentration of the contaminant of concern in the first phase (mg/l)
V_2 = The volume of the second phase (l)
C_2 = The concentration of the contaminant of concern in the second phase (mg/l)

7.17 The contaminant concentrations in the TCLP extract are compared to the thresholds identified in the appropriate regulations. Refer to Section 9 for quality assurance requirements.

8.0 *Procedure when volatiles are involved.*

The ZHE device has approximately a 500 ml internal capacity. Although a minimum sample size of 100 grams was required in the Section 7 procedure, the ZHE can only accommodate a maximum 100 percent solids sample of 25 grams, due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Step 8.4 provides the means of which to determine the approximate sample size for the ZHE device.

Although the following procedure allows for particle size reduction during the conduct of the procedure, this could result in the loss of volatile compounds. If possible, any necessary particle size reduction (See Step 8.5) should be conducted on the sample as it is being taken. Particle size reduction should only be conducted during the procedure if there is no other choice.

In carrying out the following steps, do not allow the waste to be exposed to the atmosphere for any more time than is absolutely necessary.

8.1 Pre-weigh the (evacuated) container which will receive the filtrate (See Section 4.6), and set aside.

8.2 Place the ZHE piston within the body of the ZHE (it may be helpful to first moisten the piston O-rings slightly with extraction fluid). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

8.3 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative subsample of the waste (25 gram maximum—See Step 8.0), record weight, and proceed to Step 8.5.

8.4 This step provides the means by which to determine the approximate sample size for the ZHE device. If the waste is liquid or multiphasic, follow the procedure outlined in Steps 7.2 to 7.9 (using the Section 7 filtration apparatus), and obtain the percent solids by dividing the weight of the solid phase of the waste by the original sample size used. If the waste obviously contains greater than 0.5% solids, go to Step 8.4.2. If it appears that the solid may comprise less than 0.5% of the waste, go to Step 8.4.1.

8.4.1 Determine the percent solids by using the procedure outlined in Step 7.10. If the waste contains less than 0.5% solids, weigh out a new 100 gram minimum representative sample, proceed to Step 8.7, and follow until the liquid phase of the waste is filtered using the ZHE device (Step 8.8). This liquid filtrate is defined as the TCLP extract, and is analyzed directly. If the waste contains greater than or equal to 0.5% solids, repeat Step 8.4 using a new 100 gram minimum sample, determine the percent solids, and proceed to Step 8.4.2.

8.4.2 If the sample is ≤ 25% solids, weigh out a new 100 gram minimum representative sample, and proceed to Step 8.5. If the sample is > 25% solids, the maximum amount of sample the ZHE can accommodate is determined by dividing 25 grams by the percent solids obtained from Step 8.4. Weigh out a new representative sample of the determined size.

8.5 After a representative sample of the waste (sample size determined from Step 8.4) has been weighed out and recorded, the sample is now evaluated for particle size (See Step 8.0). If the solid material within the waste obviously has a surface area per gram of material equal to or greater than 3.1 cm^2, or is capable of passing through a 9.5 mm (0.375 inch) standard sieve, proceed immediately to Step 8.6. If the surface area is smaller or the particle size is larger than that described above, the solid material which does not meet the above criteria is separated from the liquid phase by sieving (or equivalent means), and the solid is prepared for extraction by crushing, cutting, or grinding to a surface area or particle size as described above.

Note.—Wastes and appropriate equipment should be refrigerated, if possible, to 4°C prior to particle size reduction. Grinding and milling machinery which generates heat shall not be used for particle size reduction. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

When surface area or particle size has been appropriately altered, the solid is recombined with the rest of the waste.

8.6 Waste slurries need not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration.

8.7 Transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

Note.—If waste material has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 8.4, to determine the weight of the waste sample which will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange), and with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1–10 psi (or more if necessary) to slowly force all headspace out of the ZHE device. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

8.8 Attach evacuated pre-weighed filtrate collection container to the liquid inlet/outlet value and open valve. Begin applying gentle pressure of 1–10 psi to force the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within any 2 minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

Note.—Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

8.9 The material in the ZHE is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

Note.—Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid—but even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the TCLP extraction as a solid.

If the original waste contained less than 0.5% solids, (See Step 8.4) this filtrate is defined as the TCLP extract, and is analyzed directly—proceed to Step 8.13.

8.10 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (See Step 8.1) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (See Steps 8.13 and 8.14), or stored at 4°C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample (See Step 8.4). Record the final weight of the liquid and solid phases.

8.11 The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Section 5.6).

8.11.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the amount of fluid introduced into the device equals 20 times the weight of the solid phase of the waste that is in the ZHE.

8.11.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve, and disconnect the extraction fluid line. Check the ZHE to make sure that all valves are in their closed positions. Pick up the ZHE and physically rotate the device in an end-over-end fashion 2 or 3 times.

Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Put 5–10 psi behind the piston (if necessary), and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5–10 psi and check all ZHE fittings to insure that they are closed.

8.11.3 Place the ZHE in the rotary extractor apparatus (if it is not already there), and rotate the ZHE at 30 + 2 rpm for 18 hours. The temperature shall be maintained at 22 ± + 3°C during agitation.

8.12 Following the 18 hour extraction, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve, and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Replace ZHE O-rings or other fittings, as necessary, and redo the extraction with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag, gas-tight syringe) holding the initial liquid phase of the waste, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Step 8.8. All extract shall be filtered and collected if the extract is multi-phasic or if the waste contained an initial liquid phase.

Note.—If the glass fiber filter is not intact following agitation, the filtration device discussed in the NOTE in Section 4.3.1 may be used to filter the material within the ZHE.

8.13 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 8.12 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Step 8.12, and the initial liquid phase (Step 8.8) are collectively defined as the TCLP extract.

8.14 The TCLP extract will be prepared and analyzed according to the appropriate SW-846 analytical methods, as identified in Appendix III of 40 CFR 261. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to 0.1 ml), conduct the appropriate analyses and combine the results mathematically by using a simple volume weighted average:

$$\text{Final contaminant concentration} = \frac{(V_1)(C_1)+(V_2)(C_2)}{V_1+V_2}$$

where:

V_1 = The volume of the first phase (1)
C_1 = The concentration of the contaminant of concern in the first phase (mg/l)
V_2 = The volume of the second phase (1)
C_2 = The concentration of the contaminant of concern in the second phase (mg/l)

8.15 The contaminant concentrations in the TCLP extract are compared to the thresholds identified in the appropriate regulations. Refer to Section 9 for quality assurance requirements.

9.0 *Quality Assurance requirements.*

9.1 All data, including quality assurance data, should be maintained and available for reference or inspection.

9.2 A minimum of one blank for every 10 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment is occurring. One blank shall also be employed for every new batch of leaching fluid that is made up.

9.3 All quality control measures described in the appropriate analytical methods shall be followed.

9.4 The method of standard addition shall be employed for each waste type if: 1) Recovery of the compound from spiked splits of the TCLP extract is not between 50 and 150%, or 2) If the concentration of the constituent measured in the extract is within 20% of the appropriate regulatory threshold. If more than 1 extraction is being run on samples of the same waste, the method of standard addition need only be applied once and the percent recoveries applied to the remainder of the extractions.

9.5 TCLP extracts shall be analyzed within the following periods after generation: Volatiles—14 days, Semi-volatiles—40 days, Mercury—28 days, and other Metals—180 days.

TABLE 1.—VOLATILE CONTAMINANTS [1]

Compound	CASNO
Acetone	67-64-1
Acrylonitrile	107-13-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
1,1,1,2-Tetrachloroethane	630-20-6

TABLE 1.—VOLATILE CONTAMINANTS [1]—Continued

Compound	CASNO
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

[1] Includes compounds identified in both the Land Disposal Restrictions Rule and the Toxicity Characteristic.

TABLE 2.—SUITABLE ROTARY AGITATION APPARATUS [1]

Company	Location	Model
Associated Design and Manufacturing Co.	Alexandria, Virginia, (703) 549-5999	4-vessel device 6-vessel device.
Lars Lande Manufacturing.	Whitmore Lake, Michigan, (313) 449-4116	10-vessel device
IRA Machine Shop and Laboratory.	Santurce, Puerto Rico, (809) 752-4004	16-vessel device
EPRI Extractor		6-vessel device [2]

[1] Any device which rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

[2] Although this device is suitable, it is not commercially made. It may also require retrofitting to accommodate ZHE devices.

TABLE 3.—SUITABLE ZERO-HEADSPACE EXTRACTOR VESSELS

Company	Location	Model No
Associated Design and Manufacturing Co	Alexandria, Virginia, (703) 549-5999	3740-ZHB
Millipore Corp	Bedford, Massachusetts, (800) 225-3384	SD1P581C5

TABLE 4.—SUITABLE FILTER HOLDERS [1]

Company	Location	Model	Size (mm)
Nuclepore Corp	Pleasanton, California, (800) 882-7711.	425910 410400	142 47
Micro Filtration Systems.	Dublin, California, (415) 828-6010	302400	142
Millipore Corp	Bedford, Massachusetts, (800) 225-3384.	YT30142HW XX1004700	142 47

[1] Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic contaminants are of concern.

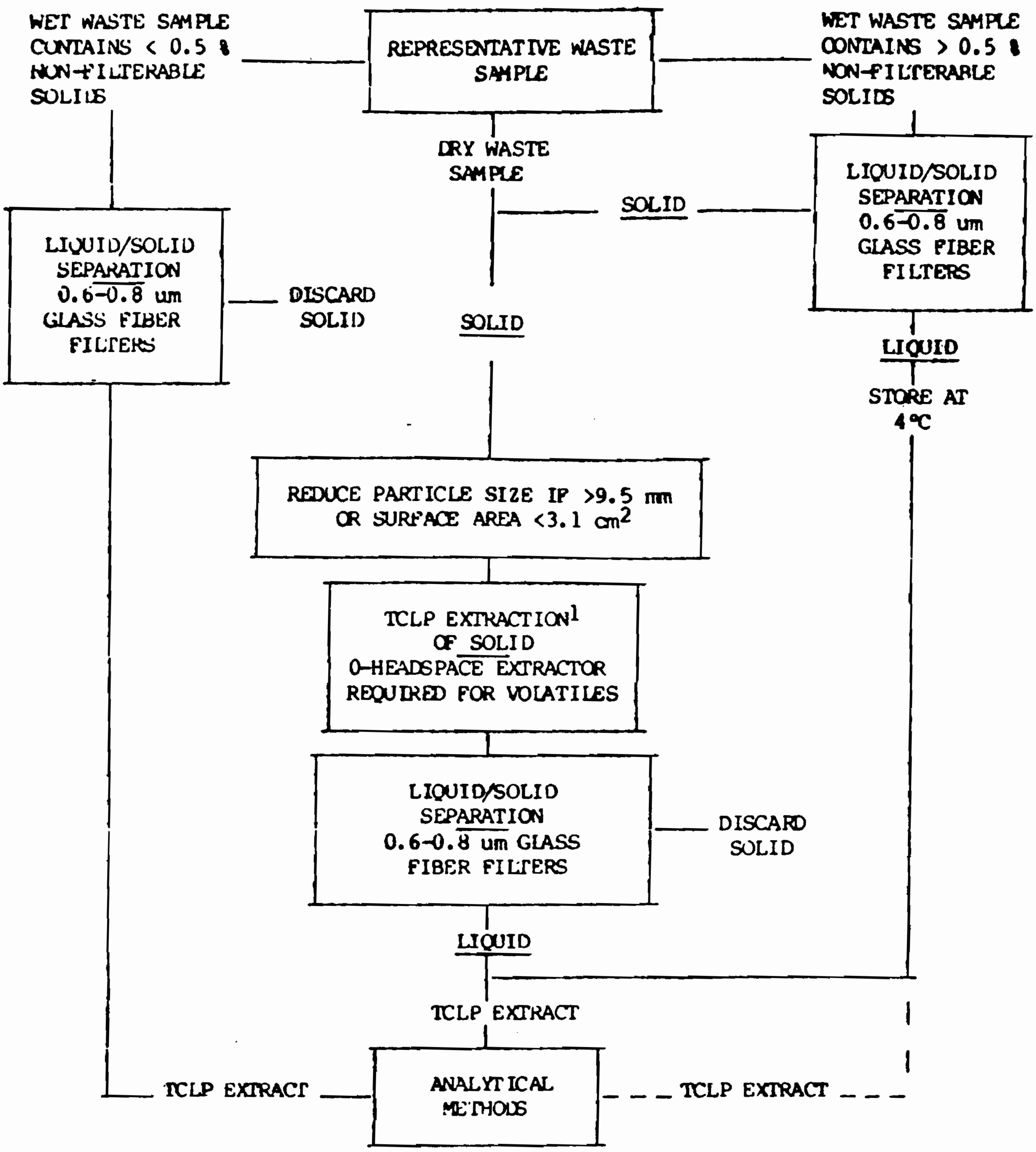
TABLE 5.—SUITABLE FILTER MEDIA

Company	Location	Model	Pore size [1]
Whatman Laboratory Products, Inc	Clifton, New Jersey (201) 773-5800.	GFF	0.7

[1] Nominal pore size

BILLING CODE 6560-50-M

FIGURE 1: TCLP Flowchart



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[1] The extraction fluid employed is a function of the alkalinity of the solid phase of the waste.

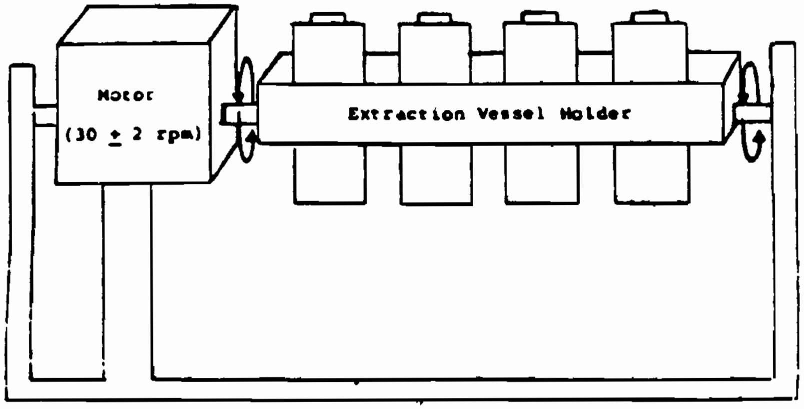

Figure 2: Rotary Agitation

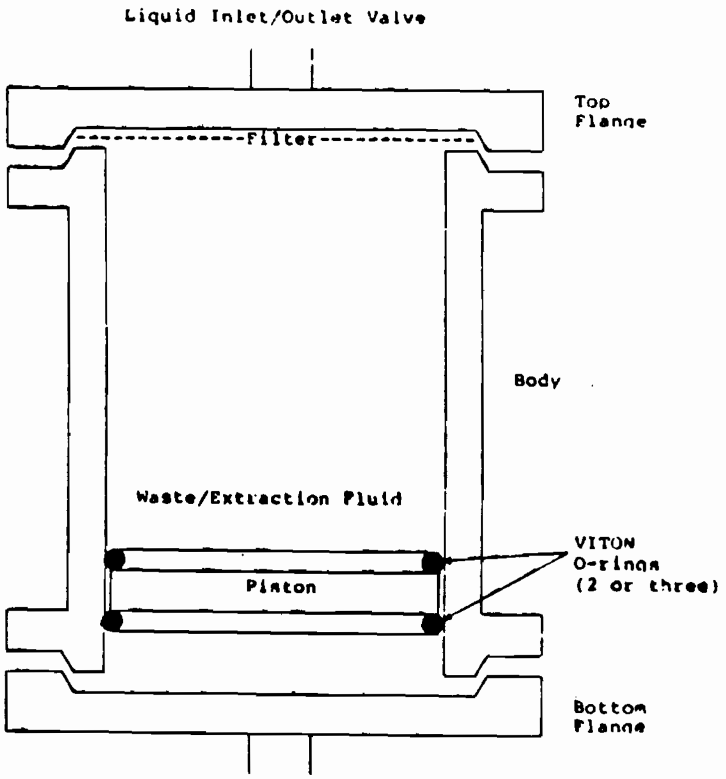

Figure 3: Zero-Headspace Extraction Vessel

BILLING CODE 6560-50-C

4. Amend Table 1 of Appendix III of Part 261 to add the following compounds and methods in alphabetical order:

Appendix III—Chemical Analysis Test Methods

* * * * *

TABLE 1.—ANALYSIS METHODS FOR ORGANIC CHEMICALS CONTAINED IN SW-846

Compound	First edition method(s)	Second edition method(s)
* * *	*	*
Benzene	8.02, 8.24	8020, 8024, 5030/8240
* * *	*	*
Bis(2-chloroethyl)ether	8.01, 8.24	8010, 8240, 3510/8270
* * *	*	*
Cresol(s)	8.04, 8.25	8040, 8250, 3510/8270
* * *	*	*
Dichlorobenzene(s)	8.01, 8.02, 8.12, 8.25	8010, 8120, 8250, 3519/8270
1,2-Dichloroethane	8.01, 8.24	8010, 8240, 6030/8240
1,1-Dichloroethylene		5030/8240
* * *	*	*
2,4-Dinitrotoluene	9.09, 8.25	8090, 8250, 3510/8270
* * *	*	*
Hexachlorobenzene	8.12, 8.26	8120, 8250, 3510/8270
Hexachlorobutadiene	9.12, 8.25	8120, 8250, 3510/8270
Hexachloroethane	8.12, 8.25	8010, 8240, 3510/8270
* * *	*	*
Isobutanol		5030/8240
* * *	*	*
Methoxychlor		8080
Methylene chloride		5030/8240
* * *	*	*
Nitrobenzene	8.09, 8.25	8090, 8250, 3510/8270

TABLE 1.—ANALYSIS METHODS FOR ORGANIC CHEMICALS CONTAINED IN SW-846—Continued

Compound	First edition method(s)	Second edition method(s)
* * *	*	*
Pentachlorophenol	8.04, 8.25	8040, 8250, 3610/8270
Phenol	8.04, 8.25 8.22	8040, 8250, 8140, 3610/8270
* * *	*	*
Pyridene	8.06, 8.09, 8.26	8090, 8250, 3510/8270
* * *	*	*
Tetrachloroethane(s)	8.01, 8.24	8010, 8240, 6030/8240
* * *	*	*
Tetrachloroethylene		5030/8240
Tetrachlorophenol(s)	8.04, 8.24	8040, 8250, 3510/8270
Toluene	8.02, 8.24	8020, 8024, 5030/8240
* * *	*	*
Trichloroethane(s)	8.01, 8.24	8010, 8240, 6030/8240
Trichloroethylene		6030/8240
* * *	*	*
Trichlorophenol(s)	8.04, 8.25	8040, 8250, 3510/8270
* * *	*	*

PART 271—REQUIREMENTS FOR AUTHORIZATION OF STATE HAZARDOUS WASTE PROGRAMS

1. The authority citation for Part 271 continues to read as follows:

Authority: Secs. 1006, 2002(a), and 3006 of the Solid Waste Disposal Act, as amended by the Resource Conservation and Recovery Act of 1976, as amended (42 U.S.C. 6905, 6912(a), and 6926).

2. Amend § 271.1 Paragraph (j) by adding the following entry to Table 1 in chronological order by date of publication:

§ 271.1 Purpose and scope.

* * * * *

(j) * * *

TABLE 1.—REGULATIONS IMPLEMENTING THE HAZARDOUS AND SOLID WASTE AMENDMENTS OF 1984

Date	Title of regulation
* * *	* *
June 13, 1986	Toxicity Characteristic
* * *	* *

PART 302—DESIGNATION, REPORTABLE QUANTITIES, AND NOTIFICATION

1. The authority citation for Part 302 continues to read as follows:

Authority: Sec. 102 of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980, 42 U.S.C. 9602; Secs. 311 and 501(a) of the Federal Water Pollution Control Act, 33 U.S.C. 1321 and 1361.

2. Section 302.4 is amended by revising the entry for "Characteristic of EP Toxicity" in Table 302.4 and the footnotes are republished as follow:

§ 302.4 Designation of hazardous substances.

* * * * *

TABLE 3024.—LIST OF HAZARDOUS SUBSTANCES AND REPORTABLE QUANTITIES

Hazardous substance	CASRN	Regulatory synonyms	Statutory			Final RQ	
			RQ	Code†	RCRA waste No.	Category	Pounds (Kg)
*	*	*	*				
Unlisted Hazardous Wastes			1*	4			
*	*	*	*				
Toxicity Characteristic			1*	4			
Acrylonitrile	107131	2-Propenenitrile	100	1,2,4	D018	B	100=(45.4)
Arsenic			1*	4	D004	X	1=(0.454)
Barium			1*	4	D005	C	1000(454)
Benzene	71432		1000	1,2,3,4	D019	C	1000=(454)
Bis(2-chloroethyl) ether	111444	Dichloroethyl ether Ethane, 1,1'-oxybis[2-chloro-	1*	2,4	D020	X	1=(0.454)
Cadmium			1*	4	D006	X	1=(0.454)
Carbon disulfide	75150	Carbon bisulfide	5000	1,4	D021	D	5000==(2270)
Carbon tetrachloride	56235	Methane, tetrachloro-	5000	1,2,4	D022	D	5000=(2270)
Chlordane	57749	Chlordane, technical 4,7-Methanoindan, 1,2,4,5,6,7,8,8-octachloro-3a,4,7,7a-tetrahydro-.	1	1,2,4	D023	X	1=(0.454)
Chlorobenzene	108907	Benzene, chloro-	100	1,2,4	D024	B	100(45.4)
Chloroform	67663	Methane, trichloro-	5000	1,2,4	D025	D	5000=(2270)
Chromium			1*	4	D007	X	1=(0.454)
o-Cresol	95487	o-Cresylic acid	1000	1,4	D026	C	1000==(454)
m-Cresol	108394	m-Cresylic acid	1000	1,4	D027	C	1000==(454)
p-Cresol	106445	p-Cresylic acid	1000	1,4	D028	C	1000==(454)
2,4-D			100	1,4	D016	B	100(45.4)
1,2-Dichlorobenzene	95501	Benzene, 1,2-dichloro-o-Dichlorobenzene	100	1,2,4	D029	B	100(45.4)
1,4-Dichlorobenzene	106467	Benzene, 1,4-dichloro-p-Dichlorobenzene	100	1,2,4	D030	B	100(45.4)
1,2-Dichloroethane	107062	Ethane, 1,2-dichloro-Ethylene dichloride	5000	1,2,4	D031	D	5000=(2270)
1,1-Dichloroethylene	75354	Ethene, 1,1-dichloro-Vinylidene chloride	5000	1,2,4	D032	D	5000=(2270)
2,4-Dinitrotoluene	121142	Benzene, 1-methyl-2,4-dinitro-	1000	1,2,4	D033	C	1000=(454)
Endrin			1	1,4	D012	X	1(0.454)
Heptachlor (and hydroxide)	76448	4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-.	1	1,2,4	D034	X	1=(0.454)
Hexachlorobenzene	118741	Benzene, hexachloro	1*	2,4	D035	X	1=(0.454)
Hexachlorobutadiene	87683	1,3-Butadiene, 1,1,2,3,4,4- hexachloro-	1*	2,4	D036	X	1=(0.454)
Hexachloroethane	67721	Ethane, 1,1,1,2,2,2-hexachloro-	1*	2,4	D037	X	1=(0.454)
Isobutanol	78831	Isobutyl alcohol 1-Propanol, 2-methyl-	1*	4	D038	D	5000(2270)
Lead			1*	4	D008	X	1==(0.454)

APPENDIX B

CHAIN OF CUSTODY PROCEDURES

1. Definition

A sample will be considered under a person's custody if: 1) it is in a person's physical possession; 2) in view of the person after he has taken possession; 3) secured by that person so that no one can tamper with the sample; or 4) secured by that person in an area which is restricted to authorized personnel. A person who has samples under his custody must comply with the procedures described in the following sections.

2. Sample Labels

Sample labels (Figure 1) are necessary to prevent misidentification of samples. Gummed paper labels or tags will include at least the following information:

Sample number
Name of collector
Date and time of collection
Place of collection

Labels will be affixed to sample containers prior to or at the time of sampling. The labels will be filled out at the time of collection.

3. Sample Seals

Sample seals are used to detect unauthorized tampering of samples following sample collection up to the time of analysis. Gummed paper seals will be used for this purpose. The paper seal will include, at least, the following information:

Sample number (This number must be identified with the number on the sample label.)
Collector's name
Date and time of sampling

FIGURE 1

SAMPLE LABEL EXAMPLE

Collector ____Sample No. _________

Place of Collection ____

____

Date Sampled ____________________________Time Sampled ________________

Field Information ____

____

____

____

The seal will be attached in such a way that it is necessary to break it in order to open the sample container. An example of a sample seal is shown in Figure 2. Seals will be affixed to containers before the samples leave the custody of the sampling personnel.

4. Field Log Book

All information pertinent to a field survey or sampling will be recorded in a log book. This will be bound, preferably with consecutively numbered pages that are 21.6 by 27.9 cm (8.5 by 11 in.). As a minimum, entries in the log book will include the following:

Purpose of sampling (e.g., surveillance, contract number)

Location of sampling point

Name and address of field contact

Producer of waste and address, if different than location

Type of process (if known) producing waste

Type of waste (e.g., sludge, wastewater)

Suspected waste composition, including concentrations

Number and volume of sample taken

Description of sampling point and sampling methodology

Date and time of collection

Collector's sample identification number(s)

FIGURE 2

SAMPLE SEAL EXAMPLE

NAMES AND ADDRESS OF ORGANIZATION COLLECTING SAMPLES

Person Collecting Sample ____________________________ Sample No. ________
(signature)

Date Collected ______________________________ Time Collected __________
Place Collected ____
____
____

Sample distribution and how transported (e.g., name of laboratory, UPS, Federal Express)

References such as maps or photographs of the sampling site

Field observations

Any field measurements made (e.g., pH, flammability, explosivity)

Signatures of personnel responsible for observations

The log book will be protected and kept in a safe place.

5. Chain-of-Custody Record

To establish the documentation necessary to trace sample possession from the time of collection, a chain-of-custody record will be filled out and accompany every sample. A chain-of-custody record is illustrated in Figure 3.

The record will contain the following minimum information.

Sample number
Signature of collector
Date and time of collection
Place and address of collection
Waste type
Signature of persons involved in the chain of possession
Inclusive dates of possession

6. Sample Analysis Request Sheet

The sample analysis request sheet (Figure 4) will accompany the sample on delivery to the laboratory. The filed portion of this form will be completed by the person collecting the sample and

CHAIN OF CUSTODY RECORD

ROJECT NO | PROJECT NAME

ABORATORY NAME:

AMPLE I.D NO.	DATE	TIME	COMP	GRAB	SAMPLE LOCATION / SAMPLING METHOD	NO. OF CON-TAINERS	ANALYSIS											REMARKS SAMPLE DESCRIPTION & OTHER ANALYSES

hipped Via:

elinquished by (Signature) rinted Name	Date/Time	Agent of:	Rec'd. by (Signature) Printed Name	Date/Time	Agent of:
elinquished by (Signature) rinted Name	Date/Time	Agent of:	Rec'd. by (Signature) Printed Name	Date/Time	Agent of:
elinquished by (Signature) rinted Name	Date/Time	Received for Laboratory by: (Signature) Printed Name	Date/Time	Remarks:	
ampler (Signature)		Samplers Name (Print)			

Figure 4

SAMPLING ANALYSIS REQUEST

PART I: Field Section

Collector ____________ Date Sampled ________ Time ________ hours

Affiliation of Sampler ______________________

Address ______________________
number street city state zip

Telephone (__)__________ Company Contact ______________

LABORATORY SAMPLE NUMBER	COLLECTOR'S SAMPLE NO.	TYPE OF SAMPLE*	FIELD INFORMATION**

Analysis Requested ______________________

____

____

Special Handling and/or Storage ______________________

____

PART II: LABORATORY SECTION**

Received by ______________ Title __________ Date ________

Analysis Required ______________________

____

* Indicate whether sample is soil, sludge, etc.
**Use back of page for additional information relative to sample location.

Figure 4. Example of hazardous waste sample analysis request sheet.

will include most of the pertinent information noted in the log book. The laboratory portion of this form will be completed by laboratory personnel and to include at a minimum:

Name of person receiving the sample
Laboratory sample number
Date of sample receipt
Sample allocation
Analyses to be performed

7. Sample Delivery to the Laboratory

The sample will be delivered to the laboratory for analysis as soon as practicable - immediately to the laboratory or usually within one or two days after sampling to the contracted laboratory. The sample will be accompanied by the chain-of-custody record (Figure 3). The sample will be delivered to the person in the laboratory authorized to receive samples (often referred to as the sample custodian).

8. Receipt and Logging of Sample

In the laboratory, a sample custodian will be assigned to receive the samples. Upon receipt of a sample, the custodian will: inspect the condition of the sample and the sample seal; reconcile the information on the sample label and seal against that on the chain-of-custody record; assign a laboratory number; log in the sample in the laboratory log book; and store the sample in a secured sample storage room or cabinet until assigned to an analyst for analysis.

The sample custodian will inspect the sample for any leakage from the container. A leaky container containing multiphase sample will not be accepted for analysis, if the sample is contained in a plastic bottle and the container walls show that the sample is under pressure or releasing gases, the sample will be treated with caution since it may be explosive or release extremely poisonous gases. The custodian should examine whether the sample seal is intact or broken. Any discrepancies between the information on the sample label and seal and the information that is on the chain-of-custody record and the sample analysis request sheet will be resolved before the sample is assigned for analysis. Results of the inspection will be noted on the sample analysis request sheet and on the laboratory sample log book.

Incoming samples will carry the inspector's or collector's identification numbers. To further identify these samples, the laboratory will assign its own identification numbers, which normally are given consecutively. Each sample will be marked with the assigned laboratory number. This number will be correspondingly recorded on a laboratory sample log book along with the information describing the sample. The sample information will be copied from the sample analysis request sheet and cross-checked against that on the sample label.

9. Assignment of Sample for Analysis

The laboratory supervisor will assign the sample for analysis. The supervisor will review the information on the sample analysis request sheet, which includes inspection notes recorded by the laboratory sample custodian. The technician assigned to analysis will record in the laboratory notebook the identifying information about the sample, the date of receipt, and other pertinent information. This record will also include the subsequent testing data and calculations.

Once the sample has been received in the laboratory, the supervisor or his assignee will be responsible for its care and custody.

APPENDIX C

QUALITY CONTROL REQUIREMENTS FOR ANALYSES OF METALS BY ATOMIC ABSORPTION

1.0 Minimum Requirements:

1.1 All quality control data should be maintained and available for easy reference or inspection.

1.2 An unknown performance sample (when available) must be analyzed once a year for the metals measured. Results must be within the control limit established by EPA. If problems arise, they should be corrected, and a follow-up performance sample should be analyzed.

2.0 Minimum Daily Control:

2.1 After a calibration curve composed of a minimum of a reagent blank and three standards have been prepared, subsequent calibration curves must be verified by use of at least a reagent blank and one standard at or near the midpoint standard. Daily checks must be within ± 10% of original curve.

2.2 If 10 or more samples per day are analyzed, the working standard curve must be verified by running an additional standard at or near the midpoint standard every 10 samples. Checks must be within ± 10% of original curve.

3.0 Optional Requirements

3.1 A current service contract should be in effect on balances and the atomic absorption spectrophotometer.

3.2 Class S weights should be available to make periodic checks on balances.

3.3 Chemicals should be dated upon receipt of shipment and replaced as needed or before shelf life has been exceeded.

3.4 A known reference sample (when available) should be analyzed once per quarter for the metals measured. The measured value should be within the control limits established by EPA.

3.5 At least one duplicate sample should be run every 10 samples, or with each set of samples to verify precision of the method.

3.6 Standard deviation should be obtained and documented for all measurements being conducted.

3.7 Quality control charts or a tabulation of mean and standard deviation should be used to document validity of data on a daily basis.

APPENDIX D

SITE HISTORY
AND
PRIOR UNDERSLAB AND SOIL SAMPLING

1. Site History

Duracell's former battery manufacturing plant is located on a 1.6 acre parcel of property at the intersection of Elm Street and Andrews Lane in North Tarrytown, New York. A plan of this parcel is presented in Drawing No. 5.

The plant consists of six buildings and the Hazardous Waste Storage Facility which were constructed at various times and the estimated construction date of each building is indicated on the Drawing. In 1945 P.R. Mallory, the predecessor company of Duracell, purchased the property with Buildings 1, 2 and 3. During the time of battery manufacturing, Mallory/Duracell constructed Building 4 in 1952, and Buildings 5 and 6, and the Hazardous Waste Storage Facility thereafter.

As part of the site investigation commissioned by Duracell in 1985, on-site soils were sampled. The methods and results of the investigation were presented in "Engineering Report Evaluating On-Site Residues" prepared by Eder Associates Consulting Engineers, P.C., October, 1985. Some of the soil sampling locations are indicated on the Drawing, designated by the prefix BH, and analysis results are presented in Table 1.

In August 1986, soils beneath floor slabs of the buildings were sampled. Sampling locations are indicated on the Drawing, designated by the prefix C, and the analysis results are presented in Table 2.

2. Underslab Soil Sampling and Analysis Methods

In July 1986, holes were drilled through the floor slabs of the buildings at the locations indicated on the Drawing and in August, 1986, samples of the soil beneath the building were obtained.

DURACELL INC.
NORTH TARRYTOWN

TABLE 1

ON-SITE SOIL METAL CONTENT

Sample Location	Metal Content 0-2 ft. Depth Below Grade Lead	 Mercury	 5-7 ft. Depth Below Grade Lead	 Mercury
BH-2	44.0	18.3	--	--
BH-15	6.8	5.4	--	--
BH-16	65.6	151.	< 2.5	0.13
BH-17	39.2	12.0	--	--
BH-20	37.8	34.5	< 2.5	3.1

NOTE: Units are mg/kg

DURACELL INC.
NORTH TARRYTOWN

TABLE 2

UNDERSLAB SOIL METAL CONTENT

	Metal Content			
Sample	0-6 in. Depth		12-18 in. Depth	
Location	Lead	Mercury	Lead	Mercury
C-1	BDL	13.3	--	--
C-2	BDL	2.5	BDL	3.7
C-3	BDL*	11.2	--	--
C-5	BDL	2.0	84.	4.4
C-6	BDL	1.0	BDL	2.0
C-7	BDL	14.	103.	67.
C-8	56.	83.	103.	21.
C-9	BDL	9.2	BDL*	23.
C-10	BDL	1.5	--	--
C-11	127.	80.	306.	16.
C-12	84.	23.	--	--

Notes:

Units are mg/kg as received.
*Average of duplicate analysis.
BDL: Below detectable limits.

Separate samples from 0-6, 6-12, 12-18, and 18-24 inches below the floor slab were collected at each location wherever possible, designated by the suffix letter A, B, C and D, respectively. A two foot long split spoon sampler was used to obtain all the samples except C-5C and C-8C, which were obtained with a soil tube. The sampler was driven into the ground, withdrawn and the sequential six inch samples were segregated into separate sample bottles starting with the lowest depth sample using new plastic spoons to segregate each sample. Prior to each use, the sampling equipment was washed with an alconox solution, rinsed with tap water and finally rinsed with distilled water obtained from Electrified Water Company of East Orange, New Jersey. All the samples were collected in new mason type glass jars covered with saran wrap and secured with metal caps.

The samples were delivered to Consolidated Technology Inc., Irvington, New York for analysis of the 0-6 (A) and 12-18 (C) inch deep samples and the laboratory report is presented at the end of this Appendix. Analyses were performed in accordance with "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-070, March 1983. Samples were digested in accordance with Section 200, Part 4.1.3 and analyzed for lead using EPA method 239.1 with atomic absorption by direct aspiration. Analysis procedures for mercury were EPA method 245.1 with atomic absorption by the manual cold vapor technique. The laboratory followed Quality Assurance

Procedures described in "Test Methods For Evaluating Solid Waste" SW-846, U.S. Environmental Protection Agency, April 1984. These procedures include duplication of sample analysis and analysis of spiked samples and blanks.

3. Underslab Soil Composition

Sample Locations C-5, C-6, C-10 and C-11 are within the envelope of buildings existing when Mallory purchased the property. Soil mercury concentrations are less than 4.4 mg/kg, except at Location C-11 where the concentration exceeds remediation levels.

Sample Locations C-1, C-2 and C-3 are within Building 4, the first building constructed by Mallory/Duracell. Soil mercury concentrations are less than 13.3 mg/kg.

Sample Location C-8 is within Building 5, constructed in 1957. Soil mercury concentration 0 to 6 inches deep is above the remediation levels.

Sample Locations C-7 and C-9 are within Building 6 which was also constructed in 1957. At both locations, the soil mercury concentrations increased with depth and exceeded remediation levels at Location C-7. Mercury concentrations at adjacent on-site soil sampling locations BH-2 and BH-17 were low.

Sample Location C-12 is within the Hazardous Waste Storage Facility, close to on-site sampling Location BH-16, and near Locations BH-15, BH-17 and BH-20. On-site soil mercury concentrations at these locations are less than 34.5 mg/kg except at BH-16 where the mercury concentration from 0-2 feet below grade exceeded the remediation levels. BH-16 is within a small strip of non-paved ground along the south wall of the facility whereas the other locations are either beneath a structure or paving. The relatively high mercury concentration, at BH-16 may be a localized phenomenon.

Consolidated Technology, Inc.
P. O BOX 261 · MT. KISCO. NEW YORK 10549 · (914) 591-9010

Job: Eder - Duracell
Sample Date: Aug. 6-14, 1986
Date Receive: Aug. 6 & 14, 1986
Time-6:15 pm

I. Results:

Sample ID	Lead T. Pb mg/kg wet	Mercury T. Hg mg/kg wet
8/6 S100A	284.0	10.47
S100C	54.9	1.53
S102A	349.4	81.27
S102C	29.6	9.68
S103A	131.9	26.96
S103C	97.1	11.40
S105A	245.8	8.65
S105C	120.6	6.58*
S106A	164.2	0.25
S106C	33.9	0.65
S108A	449.5	98.8
S108C	137.0	73.8
S109A	512.3	1518
S109C	398.1	765
8/14 C1A	ND	13.30
C2A	ND	2.47
C2C	ND	3.66
C3A	ND	11.24

Job: Eder - Duracell cont'

Sample Date: Aug. 8-14, 1986

Sample ID	Lead T. Pb mg/kg wet	Mercury T. Hg mg/kg wet
C5A	ND	1.95
C5C	83.6	4.39
C6A	ND	0.97
C6C	ND	2.03
C7A	ND	13.98
C7C	102.8	67.40
C8A	55.5	82.50
C8C	103.3	21.20
C9A	ND	9.22
C9C	ND	22.80
C10A	ND	1.49
C10C	66.9	5.18
C11A	127.4	80.40
C11C	306.3	15.80
C12A	84.3	22.6

Job: Eder - Duracell cont'
Sample Date: Aug. 6-14, 1986

Sample ID	Lead T. Pb mg/kg wet	Mercury T. Hg mg/kg wet
II. Quality Control Data:		
A. Duplicates:		
S105C	120.6	6.08 7.08
C3A	ND	
C9C	ND	
B. Spiked Data:		
20.0 ml sample - S109C	103.9%	86.76%
2.0 ml 20ppm Pb C12A	102.2%	114.6%
2.0 ml 10ppm Hg		
C. EPA 284-1 µg/l	52.6	0.39
(Pb 33.7-53.7 ")		
(Hg 0.3-1.1 ")		

III. Remarks:

Spiked samples: Pipet 20.0 ml of sample, add 2.0ml of 20.0 mg/l Lead and 2.0 ml of 10.0 mg/l Mercury to make a total volume of 24.0 ml, then run the test as a sample. %Recovery =(Actural result/Theoretical)x100.

APPENDIX E

AIR SAMPLING METHODOLOGY

APPENDIX E

This appendix presents air sampling and analysis methods for dust, lead and mercury as presented in "NIOSH Manual of Analytical Methods" Third Edition February, 1984.

DEFINITION: Total aerosol mass

NUISANCE DUST, TOTAL

METHOD: 0500
ISSUED: 2/15/84

OSHA: 15 mg/m^3
NIOSH: no standard
ACGIH: 10 mg/m^3, total dust less than
1% quartz

PROPERTIES: quartz less than 1% [1]

SYNONYMS: boron oxide (CAS #1303-86-2) and nuisance dusts [1] including alumina (CAS #1344-28-1), calcium carbonate (CAS #1317-65-3), cellulose (paper fiber; CAS #9004-34-6), glycerin mist (CAS #56-81-5), limestone (CAS #1317-65-3), etc.

SAMPLING

SAMPLER: FILTER
(tared 37-mm, 5-µm PVC filter)

FLOW RATE: 1.5 to 2 L/min

VOL-MIN: 25 L @ 15 mg/m^3
-MAX: 133 L @ 15 mg/m^3

SHIPMENT: routine

SAMPLE STABILITY: indefinitely

BLANKS: 2 field blanks per 10 samples

BULK SAMPLE: none required

ACCURACY

RANGE STUDIED: 8 to 28 mg/m^3

BIAS: not significant

OVERALL PRECISION (s_r): 0.056 [2]

MEASUREMENT

TECHNIQUE: GRAVIMETRIC (FILTER WEIGHT)

ANALYTE: airborne particulate material

BALANCE: 0.01 mg sensitivity or better; use same balance before and after sample collection

CALIBRATION: National Bureau of Standards Class M weights

RANGE: 0.3 to 2 mg per sample

ESTIMATED LOD: 0.2 mg per sample

PRECISION: 0.08 mg per sample [3]

APPLICABILITY: The working range is 3 to 20 mg/m^3 for a 100-L air sample. This method is nonspecific and determines the total dust concentration to which a worker is exposed. It may be applied, e.g., to gravimetric determination of fibrous glass [4] in addition to the other ACGIH nuisance dusts [1].

INTERFERENCES: Organic and volatile particulate matter may be removed by dry ashing [4].

OTHER METHODS: This method is similar to the criteria document method for fibrous glass [4] and Method 5000 for carbon black. This method replaces Method S349 [5]. Impingers and direct-reading instruments may be used to collect total dust samples, but these have limitations for personal sampling.

EQUIPMENT:

1. Environmental chamber at constant temperature and humidity (e.g., 20 °C ± 0.3 °C and 50% ± 5% RH).
2. Sampler: 37-mm PVC, 2- to 5-µm pore size membrane or equivalent hydrophobic filter and cellulose supporting pad in 37-mm cassette filter holder.
3. Personal sampling pump, 1.5 to 2 L/min, with flexible connecting tubing.
4. Microbalance, capable of weighing to 0.01 mg.
5. Vacuum desiccator.
6. Static neutralizer: e.g., Po-210; replace nine months after the production date.

SPECIAL PRECAUTIONS: None.

PREPARATION OF FILTERS BEFORE SAMPLING:

1. Dry filters and backup pads under vacuum in the vacuum desiccator for at least 15 min.
2. Release the vacuum, remove the desiccator cover and equilibrate the filters in the environmental chamber for at least 1 hr.
3. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
4. Weigh the filters in the environmental chamber. Record the filter tare weight, W_1 (mg).
   a. Zero the balance before each weighing.
   b. Handle the filter with forceps (nylon forceps if further analyses will be done).
   c. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
5. Place the weighed filters on top of the backup pads in the filter cassette bottom sections and allow to stand an additional 8 to 16 hrs in the environmental chamber.
6. Reweigh the filters. If this tare weight differs by more than 0.01 mg from the first tare weight obtained in step 4 above, discard the filter.
   NOTE: Insert a rod through the outlet hole of the filter cassette bottom section to raise the backup pad and filter so that the filter can be grasped with forceps.
7. Assemble the filter in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry and mark with the same number as the backup pad.

SAMPLING:

8. Calibrate each personal sampling pump with a representative sampler in line.
9. Sample at 1.5 to 2 L/min. Do not exceed a total filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

10. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
11. Remove the top and bottom plugs from the filter cassette. Place the filter cassettes in a vacuum desiccator under vacuum for at least 15 min, followed by equilibration for at least 1 hr in the environmental chamber.
12. Remove the cassette band, pry open the cassette and remove the filter. Handle the filters very gently by the edge to avoid loss of dust.

NOTE: If the filter sticks to the underside of the cassette top, very gently lift away by using the dull side of a scalpel blade. This must be done carefully or the filter will tear.

CALIBRATION AND QUALITY CONTROL:

13. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Maintain and calibrate the balance with National Bureau of Standards Class M weights.
14. Take two to four replicate samples for every batch of field samples for quality assurance on the sampling procedures. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [6] or in the field.. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. The relative standard deviation calculated from these replicates should be recorded on control charts and action taken when the precision is out of control.

MEASUREMENT:

15. Weigh each filter, including field blanks. Record this post-sampling weight, W_2 (mg), beside its corresponding tare weight. Record anything remarkable about a filter (e.g., overload, leakage, wet, torn, etc.).

CALCULATIONS:

16. Calculate the concentration of total nuisance dust, C (mg/m³), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) + B}{V} \cdot 10^3, \ \text{mg/m}^3$$

where: W_1 = tare weight of filter before sampling (mg)
W_2 = post-sampling weight of sample-containing filter (mg)
B = mean change in field blank filter weights between tare and post-sampling (mg) (+ or -).

EVALUATION OF METHOD:

Lab testing with blank filters and generated atmospheres of carbon black was done at 8 to 28 mg/m³ [2,6]. Precision and accuracy data are given on page 0500-1.

REFERENCES:

[1] TLVs - Threshold Limit Values for 1983-84, Appendix D, ACGIH, Cincinnati, OH (1983).
[2] This Manual, Method 5000.
[3] Unpublished data from Non-textile Cotton Study, NIOSH/DRDS/EIB.
[4] NIOSH Criteria for a Recommended Standard ... Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152, 119-142 (1977).
[5] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
[6] Documentation of the NIOSH Validation Tests, S262 and S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

METHOD WRITTEN BY: Kathy Morring, Jerry Clere, and Frank Hearl, P.E., NIOSH/DRDS.

FORMULA: Pb

LEAD

METHOD: 7082
ISSUED: 2/15/84

M.W.: 207.19 (Pb); 223.19 (PbO)

OSHA: 0.05 mg/m³
NIOSH: 0.05 mg/m³ [1]
ACGIH: 0.15 mg/m³; STEL 0.45 mg/m³

PROPERTIES: soft metal;
d 11.3 g/cm³; MP 327.5 °C;
valences +2, +4 in salts

SYNONYMS: vary depending upon the chemical form (elemental lead and lead compounds except alkyl lead); CAS #1317-36-8 (PbO); CAS #7439-92-1 (Pb).

SAMPLING

SAMPLER: FILTER
(0.8-µm cellulose ester membrane)

FLOW RATE: 1 to 4 L/min

VOL-MIN: 200 L @ 0.05 mg/m³
-MAX: 1200 L

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.13 to 0.4 mg/m³ [2];
0.15 to 1.7 mg/m³ (fume) [3]

BIAS: not significant [2]

OVERALL PRECISION (s_r): 0.072 [2];
0.068 (fume) [3]

MEASUREMENT

TECHNIQUE: ATOMIC ABSORPTION, FLAME

ANALYTE: lead

ASHING: conc. HNO_3, 6 mL; 140 °C

FINAL SOLUTION: 10% HNO_3, 10 mL

FLAME: air-acetylene, oxidizing

WAVELENGTH: 283.3 nm

BACKGROUND CORRECTION: D_2 or H_2 lamp

CALIBRATION: Pb^{++} in 10% HNO_3

RANGE: 10 to 200 µg per sample [3,8]

ESTIMATED LOD: 2.6 µg per sample [9]

PRECISION (s_r): 0.03 [2]

APPLICABILITY: The working range is 0.025 to 0.5 mg/m³ for a 400-L air sample. The method is applicable to elemental lead, including Pb fume, and all other aerosols containing lead. This is an elemental analysis, not compound specific. Aliquots of the samples can be analyzed separately for additional elements.

INTERFERENCES: Use D_2 or H_2 continuum background correction to control flame or molecular absorption. High concentrations of calcium, sulfate, carbonate, phosphate, iodide, fluoride, or acetate can be corrected.

OTHER METHODS: This method combines and replaces P&CAM 173 [8] and S341 [7,9] for lead. Method 7300 (ICP-AES) is an alternate analytical method.Method 7505 is specific for lead sulfide. The following have not been revised: the dithizone method, which appears in P&CAM 102 [4] and the lead criteria document [1]; P&CAM 191 (ASV) [5]; and P&CAM 214 (graphite furnace-AAS) [6].

REAGENTS:

1. Nitric acid, conc.
2. Nitric acid, 10% (w/v). Add 100 mL conc. HNO_3 to 500 mL water; dilute to 1 L.
3. Hydrogen peroxide, 30% H_2O_2 (w/w), reagent grade.
4. Calibration stock solution, 1000 µg Pb/mL. Commercial standard or dissolve 1.00 g Pb metal in minimum volume of (1+1) HCl and dilute to 1 L with 1% (v/v) HCl. Store in a polyethylene bottle. Stable $\geq$ one year.
5. Air, compressed, filtered.
6. Acetylene.
7. Distilled or deionized water.

EQUIPMENT:

1. Sampler: Cellulose ester filter, 0.8-µm pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Atomic Absorption Spectrophotometer with an air-acetylene burner head.
4. Lead hollow cathode lamp or electrode dischargeless lamp.
5. Regulators, two-stage, for air and acetylene.
6. Beakers, Phillips, 125 mL, or Griffin, 50 mL with watchglass covers.*
7. Volumetric flasks, 10- and 100-mL.*
8. Assorted volumetric pipets as needed.*
9. Hotplate, surface temperature 140° C.
10. Bottles, polyethylene, 100-mL.

*Clean all glassware with conc. nitric acid and rinse thoroughly with distilled or deionized water before use.

SPECIAL PRECAUTIONS: Perform all acid digestions in a fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for up to 8 hrs for TWA measurements. Do not exceed a filter loading of ca. 2 mg total dust.

SAMPLE PREPARATION:

NOTE: The following sample preparation gave quantitative recovery (see EVALUATION OF METHOD) [9]. Steps 4 through 9 of Method 7300 or other quantitative ashing techniques may be substituted, especially if several metals are to be determined on a single filter.

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 3 mL conc. HNO_3, and 1 mL 30% H_2O_2 and cover with a watchglass. Start reagent blanks at this step.
   NOTE: If PbO_2 is not present in the sample, the 30% H_2O_2 need not be added [3,9].
5. Heat on hotplate (140 °C) until most of the acid has evaporated.
6. Repeat two more times using 2 mL conc. HNO_3 and 1 mL 30% H_2O_2 each time.
7. Heat on 140 °C hotplate until a white ash appears.
8. When sample is dry, rinse the watchglass and walls of the beaker with 3 to 5 mL 10% HNO_3. Allow the solution to evaporate to dryness.
9. Cool each beaker and dissolve the residues in 1 mL conc. HNO_3.
10. Transfer the solution quantitatively to a 10-mL volumetric flask and dilute to volume with distilled water.
    NOTE: If the concentration ($\underline{M}$) of any of the following is expected to exceed the lead concentration ($\underline{M}$) by 10-fold or more, add 1 mL 1 $\underline{M}$ Na_2EDTA to each flask before dilution to volume: $CO_3^=$, PO_4^{-3}, I^-, F^-, CH_3COO^-. If Ca^{++} or $SO_4^=$ are present in 10-fold excess, make all standards and samples 1% (w/w) in La^{++} [8].

CALIBRATION AND QUALITY CONTROL:

11. Prepare a series of working standards covering the range 1 to 20 µg Pb/mL (1 to 200 µg Pb per sample) by adding aliquots of calibration stock solution to 100-mL volumetric flasks. Dilute to volume with 10% HNO_3. Store the working standards in polyethylene bottles and prepare fresh weekly.
12. Analyze the working standards together with the blanks and samples (steps 17 and 18).
13. Prepare a calibration graph of absorbance vs. solution concentration (µg/mL).
14. Aspirate a standard for every 10 samples to check for instrument drift.
15. Check recoveries with at least one spiked media blank per 10 samples.
16. Use method of additions occasionally to check for interferences.

MEASUREMENT:

17. Set spectrophotometer as specified by the manufacturer and to conditions on page 7082-1.
    NOTE: An alternate wavelength is 217.0 nm [10]. Analyses at 217.0 nm have slightly greater sensitivity, but poorer signal-to-noise ratio compared to 283.3 nm. Also, non-atomic absorption is significantly greater at 217.0 nm, making the use of D_2 or H_2 continuum background correction mandatory at that wavelength.
18. Aspirate standards, samples, and blanks. Record absorbance readings.
    NOTE: If the absorbance values for the samples are above the linear range of the standards, dilute with 10% HNO_3, reanalyze, and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

19. Using the measured absorbances, calculate the corresponding concentrations (µg/mL) of lead in the sample, C_s, and average media blank, C_b, from the calibration graph.
20. Using the solution volumes (mL) of the sample, V_s, and media blanks, V_b, calculate the concentration, C (mg/m³), of lead in the air volume sampled, V (L):

$$C = \frac{C_sV_s - C_bV_b}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S241 [7] was issued on October 24, 1975, and validated over the range 0.13 to 0.4 mg/m³ for a 180-L air sample, using generated atmospheres of lead nitrate [2]. Recovery in the range 18 to 72 µg Pb per sample was 98%, and collection efficiency of 0.8-µm mixed cellulose ester filters (Millipore Type AA) was 100% for the aerosols. Subsequent studies on analytical recovery of 200 µg Pb per sample gave the results [3,9]:

Species	Digestion Method	Analytical Recovery, %
Pb metal	HNO_3 only	92 ± 4
Pb metal	HNO_3 + H_2O_2	103 ± 3
PbO	HNO_3 only	93 ± 4
PbS	HNO_3 only	93 ± 5
PbO_2	HNO_3 only	82 ± 3
PbO_2	HNO_3 + H_2O_2	100 ± 1
Pb in paint*	HNO_3 only	95 ± 6
Pb in paint*	HNO_3 + H_2O_2	95 ± 6

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*Standard Reference Material #1579, U.S. National Bureau of Standards.

Additional collection efficiency studies were also done using Gelman GN-4 filters for the collection of Pb fume, which had geometric mean diameter of 0.1 µm [3]. Mean collection efficiency for 24 sampling runs at flow rates between 0.15 and 4.0 L/min was >97 ± 2%. Overall precision, s_r, was 0.072 for lead nitrate aerosol [2,7] and 0.068 for Pb fume [3,9].

REFERENCES:

[1] Criteria for a Recommended Standard...Occupational Exposure to Inorganic Lead (Revised Criteria), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-158 (1978).

[2] Documentation of the NIOSH Validation Tests, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

[3] Heavy Metal Aerosols: Collection and Dissolution Efficiencies, Final Report of NIOSH Contract 210-79-0058, W. F. Gutknecht, M. H. Ranade, P. M. Grohse, A. Damle, and D. O'Neal, Research Triangle Institute; available as Order No. PB 83-106740 from NTIS, Springfield, VA 22161 (1981).

[4] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 102, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).

[5] Ibid, P&CAM 191.

[6] Ibid, P&CAM 214.

[7] Ibid., V. 3, S341, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

[8] Ibid, V. 5, P&CAM 173, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1979).

[9] Ibid, V. 7, S341 (revised 3/25/81), U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1982).

[10] Analytical Methods for Atomic Absorption Spectrophotometry, Perkin-Elmer (1976).

METHOD REVISED BY: Mark Millson and R. DeLon Hull, NIOSH/DPSE; S341 originally validated under NIOSH Contract CDC-94-74-45; additional studies under NIOSH Contract 210-79-0058.

FORMULA: Hg

MERCURY

METHOD: 6000
ISSUED: 2/15/84

M.W.: 200.59

OSHA: 0.1 mg/m^3
NIOSH: 0.05 mg/m^3 [1]
ACGIH: 0.05 mg/m^3

PROPERTIES: liquid; d 13.55 g/mL @ 20 °C; BP 356 °C; MP -39 °C; VP 0.16 Pa (0.0012 mm Hg; 13.2 mg/m^3) @ 20 °C

SYNONYMS: quicksilver; CAS# 7439-97-6.

SAMPLING

SAMPLER: SOLID SORBENT TUBE (30 mg silvered Chromosorb P, with glass fiber prefilter)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 0.5 L
-MAX: 7 L @ 0.1 mg/m^3

SHIPMENT: routine

SAMPLE STABILITY: at least 7 days @ 25 °C [2]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.0456 to 0.18 mg/m^3 [2,4] (3-L samples)

BIAS: not significant [2,4]

OVERALL PRECISION (s_r): 0.061 [2]

MEASUREMENT

TECHNIQUE: ATOMIC ABSORPTION, FLAMELESS

ANALYTE: elemental mercury

DESORPTION: thermal (650 to 700 °C)

CARRIER GAS: air, 1.0 L/min

WAVELENGTH: 253.7 nm

CALIBRATION: aqueous Hg^{2+} spikes onto sampler

RANGE: 0.01 to 1 µg per sample [2,4]

ESTIMATED LOD: 0.001 µg per sample

PRECISION (s_r): 0.051 [2,3]

APPLICABILITY: The working range is 0.003 to 0.3 mg/m^3 for a 3-L air sample. If a glass fiber (13-mm) filter is used to prevent potential interference from mercury compounds, this filter may be analyzed for particulate Hg species [4]. This method is not affected by high humidity environments (breakthrough determined at 85% R.H.) [2].

INTERFERENCES: Chlorine and other strong oxidizing gases which attack silver (the sampling substrate), reduce the efficiency of the sampling tube but do not interfere in the measurement. Methyl mercuric chloride is partially retained on the sampler and is, therefore, a potential interferent [5].

OTHER METHODS: This method combines and replaces P&CAM 175 [4] and S199 [3].

REAGENTS:

1. Mercury standard, 1000 µg/mL. Commercially available or dissolve 1.35 g mercuric chloride in 10 mL conc. HNO_3; dilute to 1.0 L with deionized water.
2. Mercury standard, 10 µg/mL. Dilute 1.0 mL 1000 µg/mL stock solution and 1.0 mL conc. HNO_3 to 100 mL with deionized water.
3. Mercury standard, 1.0 µg/mL. Dilute 0.1 mL 1000 µg/mL stock solution and 1.0 mL conc. HNO_3 to 100 mL with deionized water.
4. Nitric acid, conc.
5. Water, deionized.

EQUIPMENT:

1. Sampler: glass tube, 2 cm long, 6 mm OD, 4 mm ID, unsealed ends containing one section of 30 mg silvered Chromosorb P (AgCP) held in place with quartz wool plugs (commercially available); preceded by a prefilter unit (13-mm glass fiber filter in cassette filter holder).
   NOTE: A 37-mm, 0.8-µm cellulose pre-ester filter may be used if determination of particulate Hg is not desired.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Thermal desorption unit (see APPENDIX).
4. Atomic absorption spectrophotometer, cold-vapor, with Hg lamp, with 2.5- to 4-cm path length gas cell.
5. Volumetric flasks, 100-mL.*
6. Micropipets, 10- to 100-µL.*
7. Tape, PTFE.
8. Gloves, lint-free.

*Clean all glassware with conc. nitric acid and rinse thoroughly with deionized water before use.

SPECIAL PRECAUTIONS: Mercury is readily absorbed by inhalation and intact skin [1]. Vacuum pump of the thermal desorption system should be vented into a fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Remove the sampler from its storage vial and attach it to a personal sampling pump with flexible tubing. Wrap the sampler tube with PTFE tape to protect the outside of the tube from contamination.
   NOTE: Handle the sampler only with clean lint-free gloves or tissues to prevent contamination.
3. Sample at 0.01 to 0.2 L/min. Do not exceed a sample volume of 7 L if the concentrations are at or above the OSHA standard.
4. Remove the PTFE tape and return the sampler to its storage vial after sampling being careful not to contaminate the outside of the tube.

SAMPLE PREPARATION: None required.

CALIBRATION AND QUALITY CONTROL:

5. Prepare at least five working standards by adding known amounts of 1 µg/mL or 10 µg/mL mercury standard covering the range 0.01 to 1 µg Hg per sample to unused AgCP sampler tubes.
6. Analyze the working standards together with the samples and blanks (steps 8 through 12).
7. Prepare calibration graph of absorbance vs. µg Hg per tube.

MEASUREMENT:

8. Set up the measurement system as described in the APPENDIX.
9. Purge the system before analyzing samples and standards by heating the first desorption section 60 sec, allowing to cool 60 sec and heating second section 30 sec.
10. Allow system to cool 1 min before analyzing samples.
11. Measure samples and standards by inserting the tube, thermally desorbing first section 60 sec, waiting 60 sec and desorbing second section for 30 sec.
    NOTE: Handle the tube only with lint-free gloves or tissues to prevent contamination.
12. Allow the system to cool at least 30 sec before removing tube and inserting the next sample.
    CAUTION: Tubes are hot when removed. Catch them in a dry beaker as they come out of the desorption unit.

CALCULATIONS:

13. Read the absorbance of the sample and calculate the mass of Hg on the samples, W (µg), and blanks from the calibration graph. Obtain an average value for the media blanks, B (µg).
14. Calculate the concentration, C (mg/m³), of mercury in air volume sampled, V (L):

$$C = \frac{W - B}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S199 [2] was issued on July 8, 1977, and validated over the range 0.046 to 0.18 mg/m³ at 760 mm Hg and 18.0 °C using a 3-L sample [6]. Overall precision, s_r, was 0.061 with average recovery of 100.7%. The mercury concentration was independently determined by theoretical calculations based on mercury vapor pressure data and flow rate. Breakthrough did not occur after sampling 3.5 hrs at 0.184 L/min (39 L) in a Hg concentration of 0.244 mg/m³ and 85% relative humidity. Six samples stored at room temperature for seven days gave results not significantly different from samples that were analyzed immediately after exposure.

REFERENCES:

[1] Criteria for a Recommended Standard...Occupational Exposure to Inorganic Mercury, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 73-11024 (1973).

[2] Backup Data Report No. S199, prepared under NIOSH Contract 210-76-0123, available as "Ten NIOSH Analytical Methods, Set 5," available as Order No. PB 287-499 from NTIS, Springfield, VA 22161 (1977).

[3] NIOSH Manual of Analytical Methods, 2nd. ed., V. 4, S199, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).

[4] Ibid, V. 5, P&CAM 175, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).

[5] Trujillo, P. E. and E. E. Campbell. Development of a Multistage Air Sampler for Mercury, Anal. Chem., 47, 1629-1634 (1975).

[6] NIOSH Research Report-Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

METHOD REVISED BY: R. DeLon Hull, NIOSH/DPSE; S199 originally validated under NIOSH Contract CDC-99-76-123.

APPENDIX:

MERCURY MEASUREMENT SYSTEM

A. Thermal Desorption Unit. With the exception of the electrical components and the loading spring, the entire thermal desorption unit is made of either quartz or Pyrex glass. Descriptions of each important part of the thermal desorption unit numbered in Figure 6000-2 are as follows:

1-2. Loading mechanism (Figure 6000-2). Made from a glass 18/7 female standard socket joint with steel spring (#1) and a plunger tube (3-mm or 4-mm OD quartz - #2). The length of the plunger is cut to reach the sample desorption section.

3. Rubber or neoprene "O" rings for holding quartz desorption tube in place.

4. Sample desorption section. Made from 8-mm ID and 5-mm ID quartz tubing. The junction between the two sizes of tubing is tapered on the inside so that sampling tube tips fit snugly against the junction.

5. Cooling vent holes. Seven 4-mm holes in the outer jacket to vent the cooling air.

6. Sampler tube. Held in position for desorption by the spring on the loading mechanism.

7. Sample desorption coils. Twenty-eight coils of 18-gauge nichrome wire around the sample desorption section.

8. Quartz wool plugs. Used to separate each section. Although the plugs should be large enough to hold the materials, they should not be packed too tightly.

9. Cupric oxide (CuO). A 40-mm section of rod-shaped CuO, retained by a dimple in the tubing and quartz wool plug placed just downstream from the sampling tubes. When heated, the CuO oxidizes organic vapors desorbed from the sampler.

10. Cooling jacket. Pyrex glass; directs the flow of cooling air and acts as electrical insulation for the heating coils.

11. Second desorption section. Five-mm ID quartz tubing extending from the first desorption section. The contents are held in place by a dimple in the glass tube.

12. Gold section desorption coils. Thirty coils of 20-gauge nichrome wire concentrated over the gold section. The gold section is a 25-mm length of powdered gold, mixed 1:1 with 20/40 mesh sea sand to prevent fusing of the gold granules and to facilitate airflow.

13. Cooling air intake. Six-mm ID glass tubing, connected to the cooling air supply.

14. Wire lead-throughs and solderless connectors. Wires enter the cooling jacket through 5-mm holes at the ends of glass nipples on the jacket with solderless connectors between the wires and the coils. The wires are held in place with a heat resistant sealant.

15. Outlet. Butt-connected with PVC tubing overseal to a glass tube which leads to the detection system. The end of the quartz tube fits through the end of the cooling jacket with minimum clearance to limit loss of cooling air.

B. Electrical Power (Figure 6000-1). The wires to the heating coils should be heavy enough (about #12) to carry the current necessary to heat the coils. Power to the heating coils is controlled by a three-position switch with an off position, an on position for the sample desorption section and on position for the gold section. Power to the switch comes from a 120 V ac Input, 20 A variable transformer set between 20 and 30 V ac. The exact setting is determined by testing the thermal desorption unit for complete desorption of mercury from the sample tube in one heating. Complete desorption usually occurs when the sample tube reaches a maximum of 650 °C. The time required for heating may vary slightly from that stated in the method (60 sec) due to the spacing between heating coils and the concentration of heat near the sample tube and/or desorption sections.

C. Carrier Air Supply. Room air is drawn by vacuum pump, attached to a ballast tank to prevent fluctuations in airflow.

Before entering the desorption unit, the air passes through a drying tube containing anhydrous $Mg(ClO_4)_2$ or other suitable drying agent. The dimensions of the drying tube and subsequent filters are not critical but should not impede airflow. Change the drying agent periodically, depending on the humidity of the air. The dried air passes through a filter containing activated charcoal and 30/60 mesh silvered Chromosorb P or Mersorb charcoal to remove organic vapors and metallic mercury before entering the thermal desorption unit.

Carrier air passes through, in order, the thermal desorption unit, the detection system, a second filter containing Mersorb charcoal which collects the mercury, a 0 to 5 L/min flowmeter, critical orifice, and into the ballast tank of the vacuum pump.

D. Cooling air. Supplied by an air compressor at a flow rate of 20 L/min. The air enters the desorption unit near the second desorption section.

E. Detection system. Mercury from the outlet of the thermal desorption unit enters the optical cell of a flameless atomic absorption spectrophotometer through a glass tube maintained at 70 °C using a heating tape. Power for the heating tape is supplied by a 120 V ac, 8 A variable transformer.

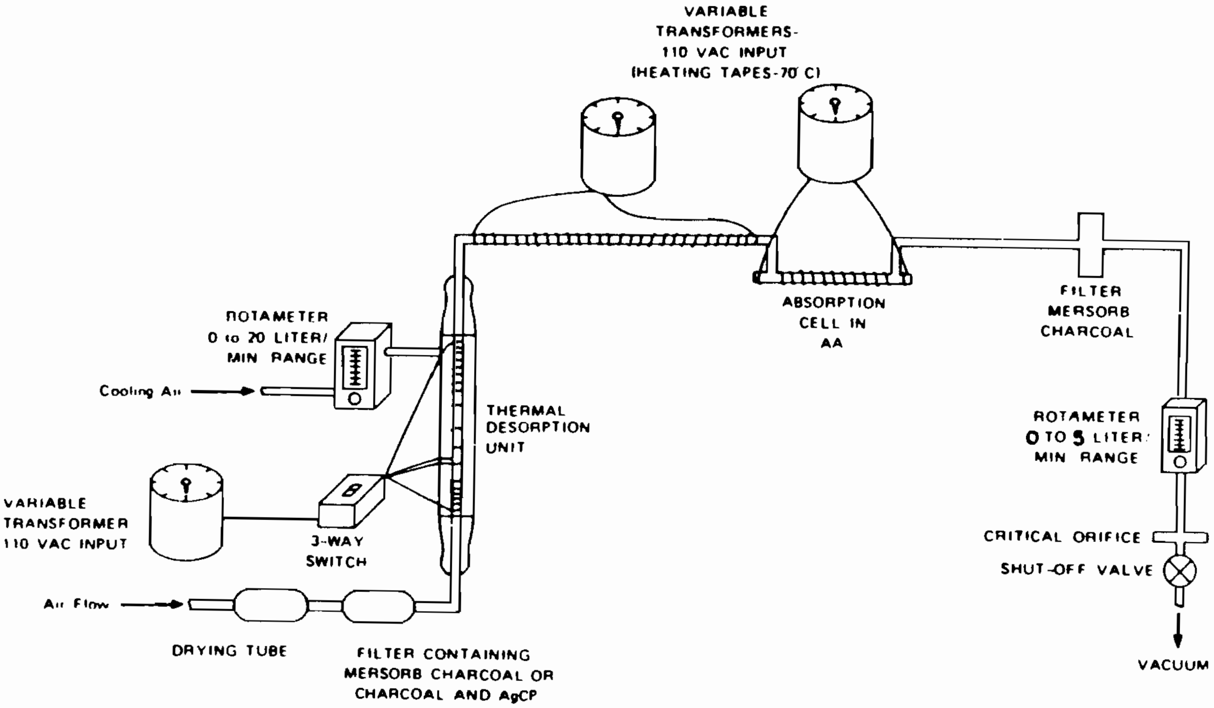
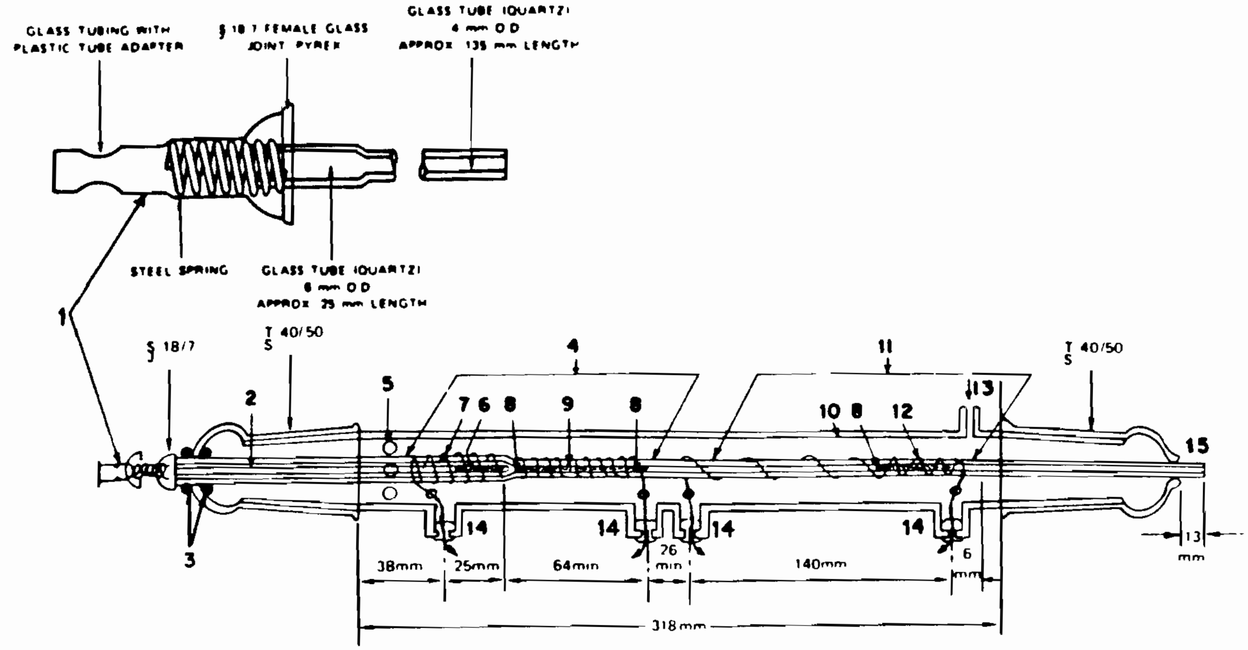
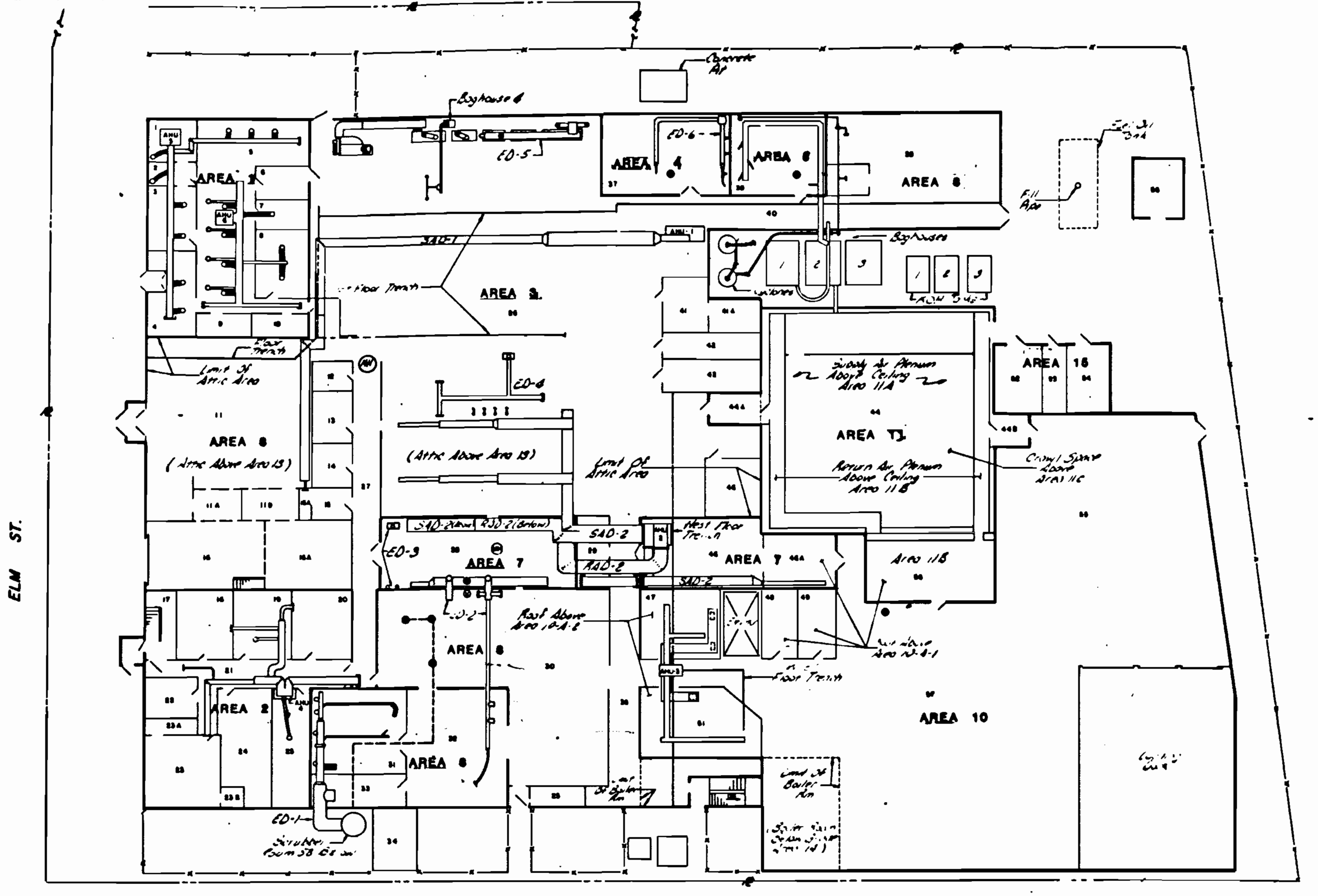


Figure 6000-1 MERCURY ANALYSIS SYSTEM

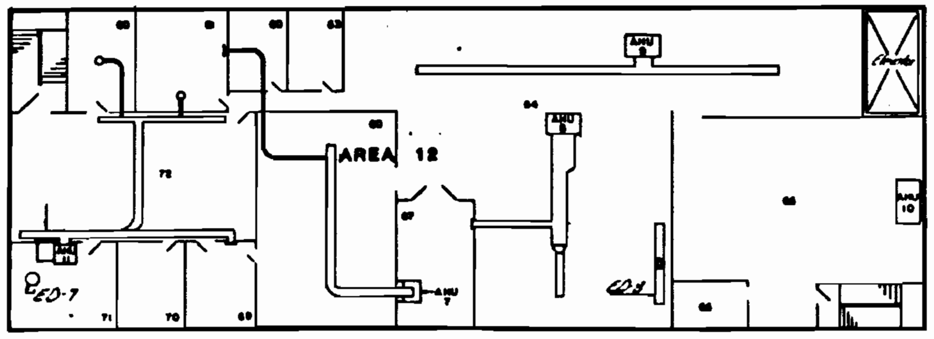


1. LOADING MECHANISM
2. QUARTZ GLASS PLUNGER TUBE
3. RUBBER "O" RINGS
4. SAMPLE DESORPTION SECTION
5. COOLING AIR VENT HOLES
6. SAMPLING TUBE
7. NICHROME WIRE COILS
8. QUARTZ GLASS WOOL PLUGS
9. CUPRIC OXIDE (CuO)
10. COOLING JACKET
11. SECOND DESORPTION SECTION
12. GOLD SECTION
13. COOLING AIR INTAKE
14. WIRE HOLES AND SOLDERLESS CONNECTORS
15. OUTLET

Figure 6000-2 THERMAL DESORPTION UNIT



FIRST FLOOR PLAN



SECOND FLOOR PLAN

LEGEND

●	Floor Drain
⊖	Manhole
⊗	Sampling Pipe
----	Below Grade Drain
AHU	Air Handling Unit
26	Room Number
SAD	Supply Air Duct
RAD	Return Air Duct
ED	Exhaust Duct

0 4 8 16 32

SCALE

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PROJECT PLAN

DURACELL INC
NORTH TARRYTOWN, NEW YORK

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TITLE
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85 FOREST AVENUE, LOCUST VALLEY, NEW YORK, 11560

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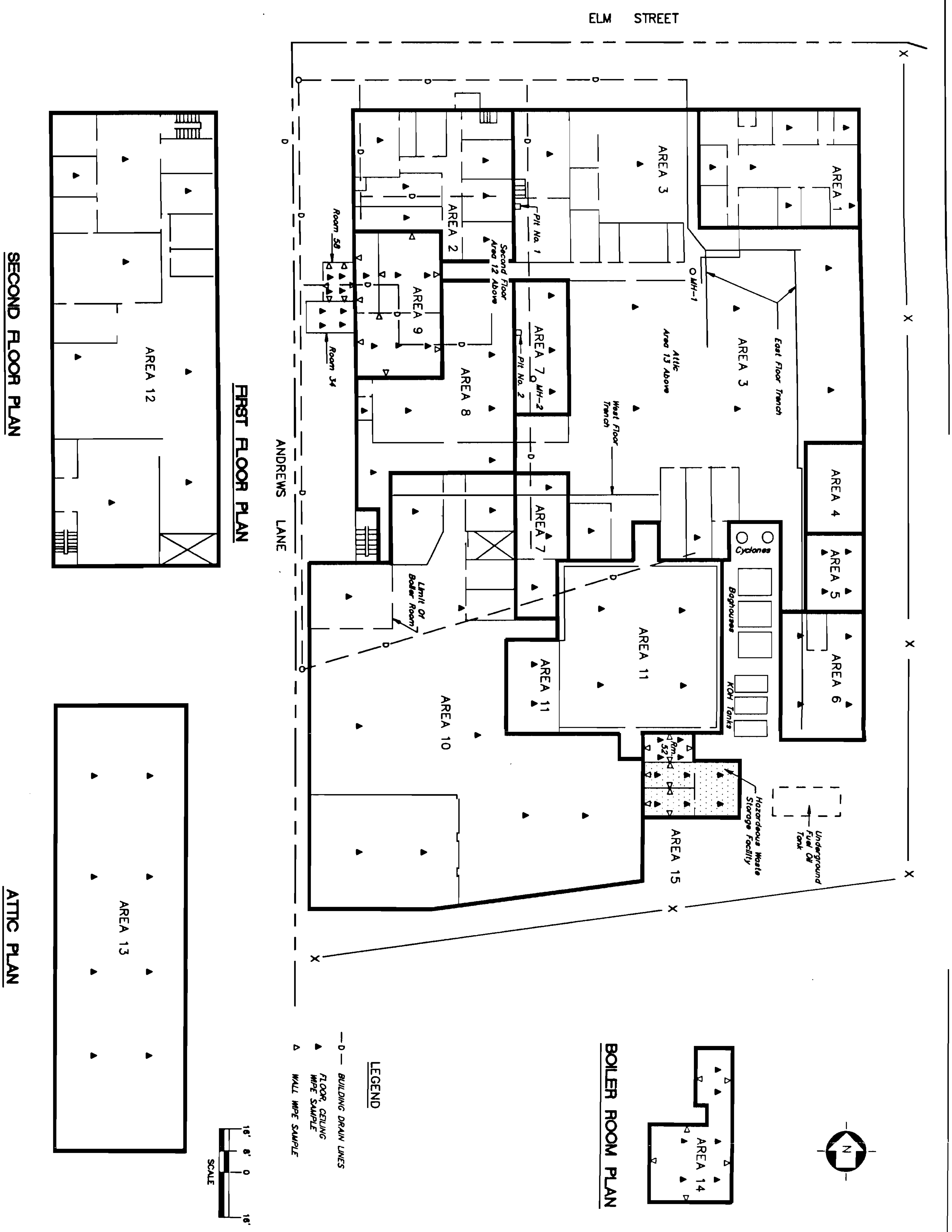
DESIGNED BY JBN	PROJECT No 425-1
APPROVED BY GAR	DWG. No 1
DATE December 1987	



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DWG. No. 2
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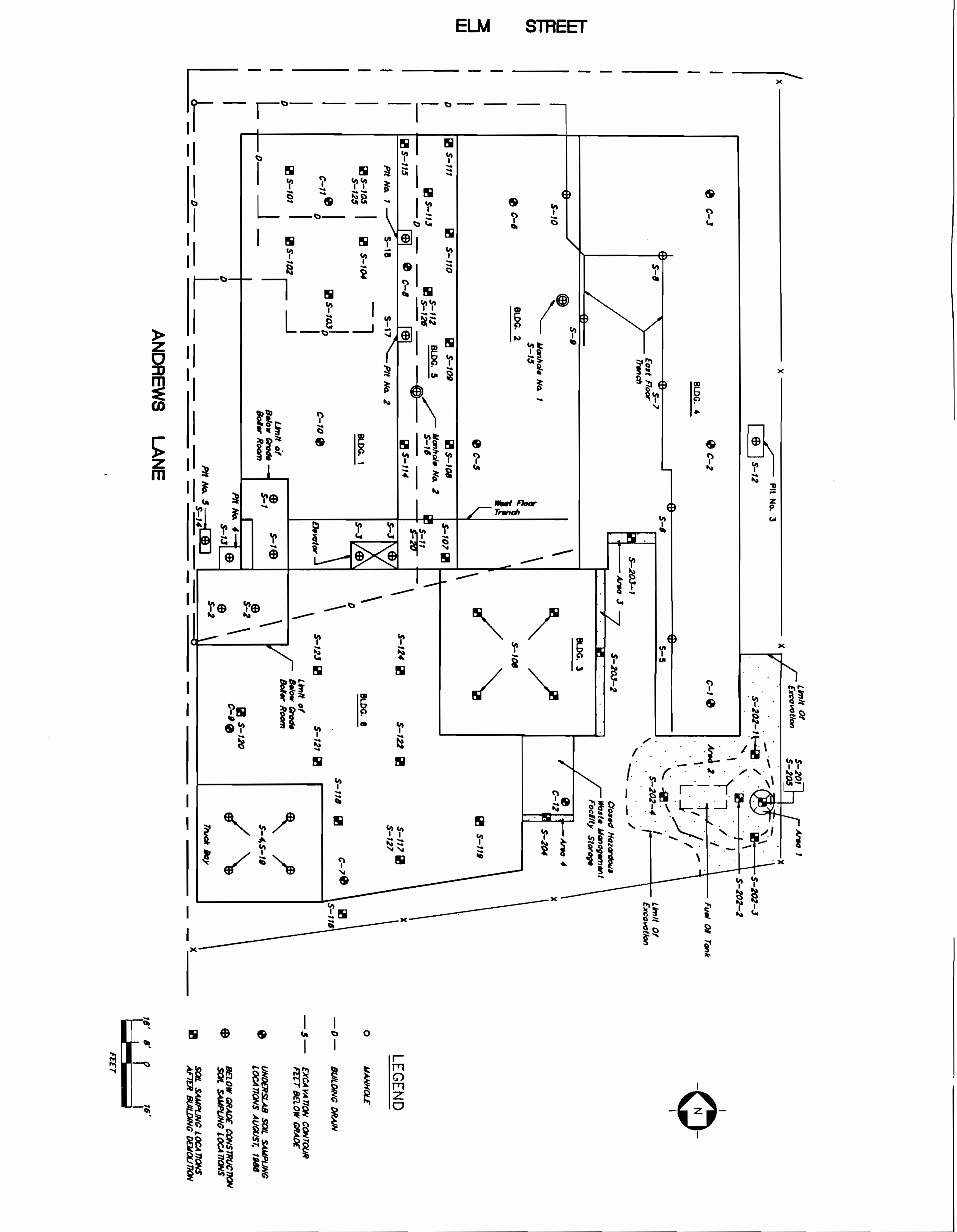

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APPROVED BY JBH	DWG. No. 3
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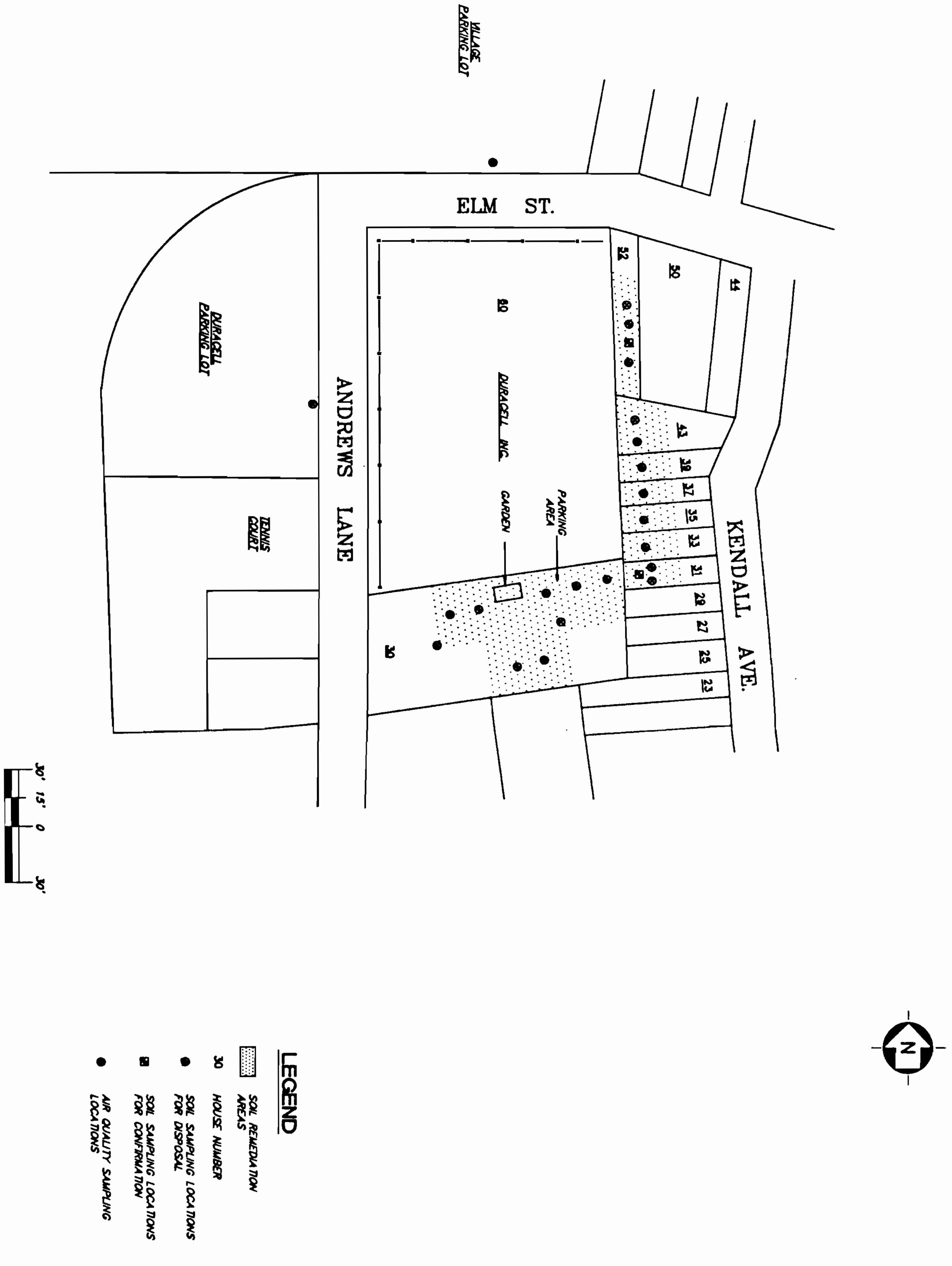

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TITLE
OFF-SITE SOIL
AND AIR SAMPLING

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2317 INTERNATIONAL LANE MADISON, WI. 53704

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SCALE 1"=30'
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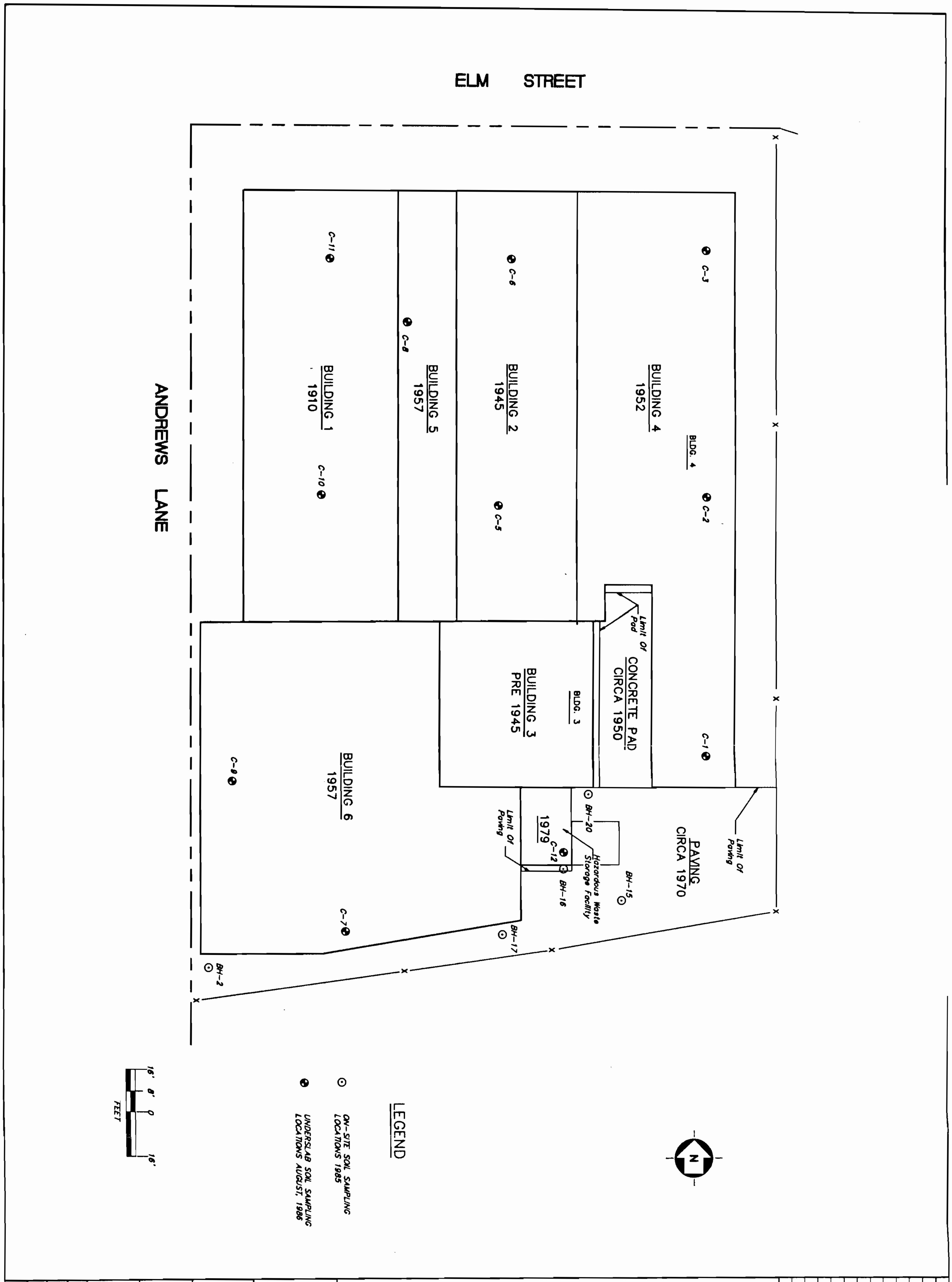
PROJECT No. 425-1
APPROVED BY JBH
DWG. No. 4
DATE December, 1987



QUALITY ASSURANCE PROJECT PLAN

DURACELL INC.

NORTH TARRYTOWN, NEW YORK

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PROJECT	QUALITY ASSURANCE PROJECT PLAN DURACELL INC. [illegible]
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	eder associates consulting engineers, p.c. 85 FOREST AVENUE LOCUST VALLEY, N.Y. 11560 2317 INTERNATIONAL LANE MADISON, WI. 53704
DRAWN BY	MSA
SCALE	1/16"=1'-0"
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PROJECT No.	425-1
APPROVED BY	GAR
DWG. No.	5
DATE	December, 1987

No.	REVISIONS	DATE	BY