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INTERIM STATUS CLOSURE PLAN FOR OLD CALCIUM CARBIDE DESULFURIZATION SLAG TREATMENT UNIT

SAGINAW NODULAR IRON CASTING PLANT
GENERAL MOTORS CORPORATION
SAGINAW, MICHIGAN

DECEMBER 1987

PRIVILEGED AND CONFIDENTIAL

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1. INTRODUCTION AND BACKGROUND

Since November 1980, the General Motors Corporation-Central Foundry Division (GMC-CFD) Saginaw Nodular Iron Plant in Saginaw, Michigan, has operated four hazardous waste units as follows:

- **Old (original) Calcium Carbide Desulfurization Slag Treatment Area.**
- Existing Calcium Carbide Desulfurization Slag Treatment Bunker.
- Paint Storage Building Drum Storage Area
- 1,1,1-Trichloroethane Hazardous Waste Control Tank

These units have operated under RCRA Interim Status regulations contained in 40 CFR Part 265 and under Michigan Act 64 hazardous waste regulations. These four units have been identified on the plant's original RCRA Part A permit application or on subsequent revisions of the Part A. Treatment in the Old (original) Desulfurization Slag Treatment Area was replaced with treatment in the Existing Desulfurization Slag Treatment Bunker. The status of the Drum Storage Area and the 1,1,1-Trichloroethane Waste Control Tank was changed to generator accumulation units in 1985. Each of the four hazardous waste management units is addressed in a separate Closure Plan.

This Closure Plan addresses the Old Calcium Carbide Desulfurization Slag Treatment Unit, which has not been in operation since the existing treatment bunker was put into operation.

Waste Generation

The GMC-CFD Saginaw Nodular Iron Plant manufactures nodular iron castings, such as differential cases, camshafts, and knuckles for automobile parts. During this manufacturing process, a hazardous waste, calcium carbide desulfurization slag, was generated and treated on-site.

The Nodular Iron Plant adds calcium carbide to remove sulfur from molten iron. This step was necessary to make nodular cast iron with appropriate metallurgical properties. The desulfurization slag formed as a reaction by-product probably contained about 1 to 3 percent unreacted calcium carbide.

When the unreacted calcium carbide in the slag came in contact with water, a reaction produced a combustible gas (acetylene). Thus, the slag was classified as a reactive hazardous waste under 40 CFR Part 261.23(a)(2). The treatment technique used to render the slag nonhazardous involved eliminating the ability of the slag to generate acetylene gas. This was accomplished by spraying the waste with water, in a waste pile and allowing the acetylene to dissipate into the atmosphere.

RCRA Part A

As identified on the original RCRA Part A application, treatment had been carried out on a concrete pad of approximately 20 feet by 20 feet and on nearby plant property. That area is known as the old (original) calcium carbide desulfurization slag treatment unit. Treatment consisted of saturating the pile of slag with water within the 20' x 20' treatment unit. Reacted slag was nonhazardous and was removed from the treatment area and disposed by landfilling.

RCRA Part B

The Saginaw Nodular Iron Plant originally filed a RCRA Part B Permit Application for a hazardous waste treatment facility (the existing treatment bunker) on February 7, 1985. This application detailed a plan where the existing bunker would be replaced with a new calcium carbide desulfurization slag treatment facility. The Part B Application included an Interim Status Closure Plan for the existing bunker. The Part B application was revised on November 5, 1985, including minor revisions to the Closure Plan for the existing bunker treatment facility.

As part of the licensing of the proposed treatment facility, GM and the MDNR attempted to negotiate a Consent Decree concerning the treatment of desulfurization slag at the Saginaw Nodular Iron Plant. While never signed, the last proposed Consent Decree, dated May 2, 1986, contained a stipulation for submittal of a Sampling and Analysis Plan for the existing bunker treatment facility to determine the environmental impact of the facility.

A draft Sampling and Analysis Plan for the existing bunker was submitted to the MDNR in June 1986. This plan was prepared for the existing calcium carbide desulfurization slag treatment bunker. The rationale of the Sampling and Analysis Plan for the existing bunker also applies to the old treatment unit. In a letter dated November 4, 1986, Mr. Donald L. Inman of the Hazardous Waste Division of the MDNR expressed his Department's approval of that plan.

Closure

GM-CFD Saginaw Nodular Iron plans to close the plant in 1988. As part of the closure, all hazardous waste management units will be closed. This Closure Plan outlines the procedures that will be used to close the Old Calcium Carbide Desulfurization Slag Treatment Unit and to document the closure activities.

40 CFR Part 265 (Interim Status standards for owners and operators of hazardous waste treatment, storage, and disposal facilities) has been incorporated by reference in its entirety under Section R299.1103(1)(m) of the Michigan Administrative Code. Therefore, all regulatory citations will be given according to the applicable portion of 40 CFR Part 265.

To facilitate review by the Michigan Department of Natural Resources (MDNR), a completed copy of MDNR's "Interim Status Land Disposal Closure and Post-Closure Checklist" has been included in Appendix A.

2. PURPOSE AND SCOPE

The purpose of this Closure Plan is to describe the closure activities that GMC-CFD Saginaw Nodular Iron (SNI) will perform to close the old (original) calcium carbide desulfurization slag treatment unit.

The scope of this document is limited to providing a Closure Plan for the old calcium carbide desulfurization slag treatment unit; other hazardous waste management units are addressed in separate closure plans. This closure plan describes the following:

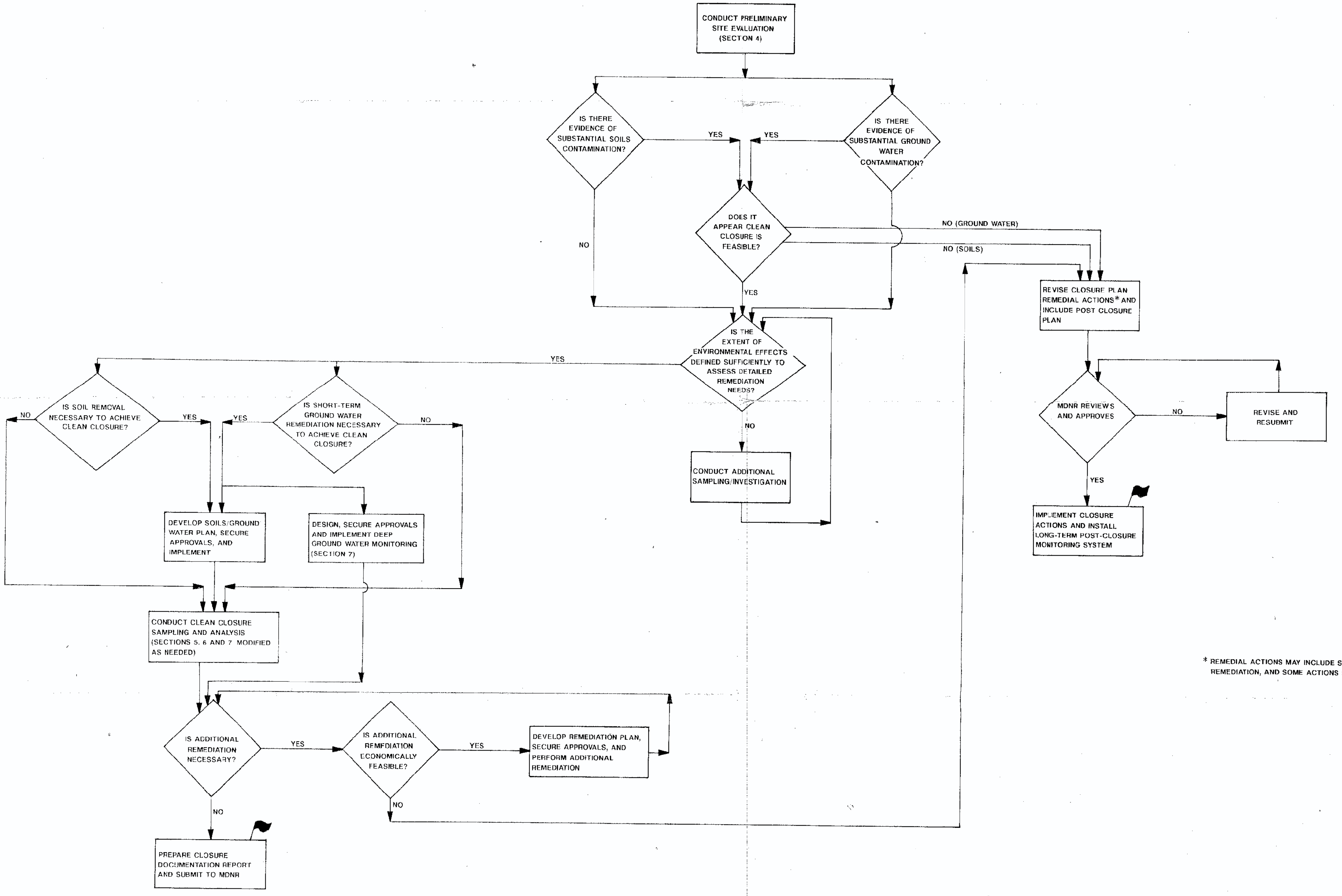
- Method of closure
- The effect closure activities will have on the need for future maintenance and the potential for post-closure release of hazardous constituents.
- Expected maximum waste inventory.
- Decontamination methods.
- Closure schedule.
- Documentation of closure activities.

This Closure Plan is intended to fulfill the closure requirements applicable to the old CaC_2 Desulfurization Slag Treatment Unit, and to describe the key activities, tests and performance standards for closure of that waste management unit. The applicable portions of 40 CFR Part 265, Subparts G and L, and Michigan Act 64 have been addressed.

The closure activities which are described in Section 5 are directed toward attaining "clean closure." If clean closure is not feasible, GMC-CFD will amend the Closure Plan to comply with post-closure requirements for the facility (40 CFR 265.117 through 40 CFR 265.120).

GENERAL MOTORS CORPORATION - CENTRAL FOUNDRY DIVISION - SAGINAW NODULAR IRON CASTING PLANT

CLOSURE DECISION DIAGRAM
FOR
INTERIM STATUS DESULFURIZATION SLAG TREATMENT UNITS



* REMEDIAL ACTIONS MAY INCLUDE SOIL REMOVALS, GROUND WATER REMEDIATION, AND SOME ACTIONS SIMILAR TO CLEAN CLOSURE.

DEC 21 1987

DIAGRAM 1

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A Preliminary Site Evaluation will be conducted before implementing the Closure Plan to determine if clean closure is feasible. Portions of that evaluation have already begun. The decision to proceed with clean closure will be made by GMC-CFD personnel based on the results of the Preliminary Site Evaluation discussed in Section 4. The closure decision diagram (Diagram 1) identifies the decision process.

3. GENERAL FACILITY INFORMATION

3.1 Facility Name, Location, and Contact

Name:

General Motors Corporation
Central Foundry Division
Saginaw Nodular Iron Plant
2100 Veterans Memorial Parkway
EPA ID Number: MID041793340
Saginaw, MI 48605-5073

Contact: William Hudson, Environmental Coordinator

3.2 Site Description

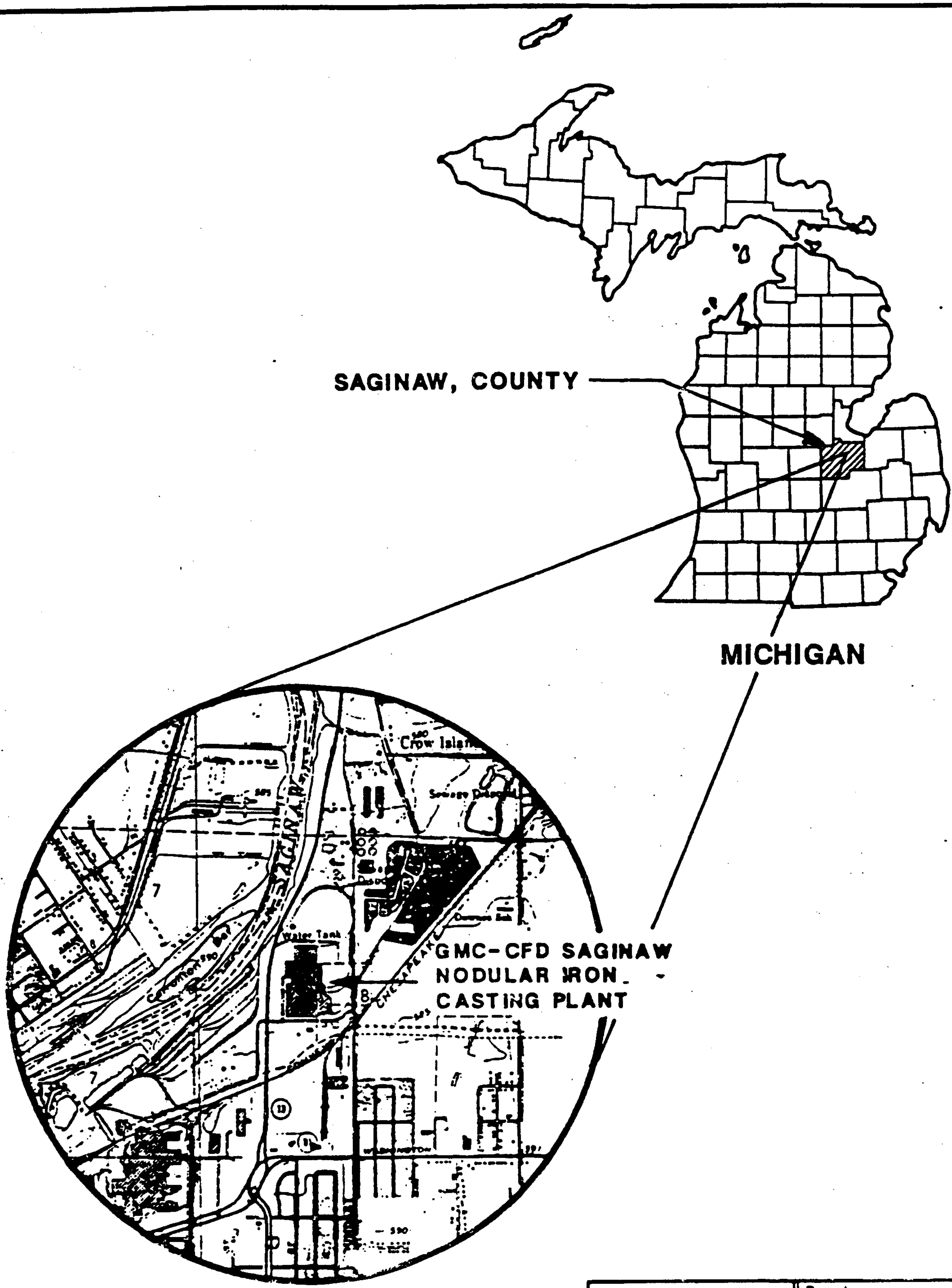
The location of the GMC-CFD Nodular Iron plant is shown in Figure 1. The old calcium carbide desulfurization slag treatment unit consisted of an area approximately 20 feet by 20 feet. The location of this treatment unit is shown in Figure 2.

3.3 Waste Characterization

The basis for classification of the calcium carbide desulfurization slag as a reactive hazardous waste was discussed in Section 1 of this Closure Plan.

The calcium carbide desulfurization slag has been characterized in a series of laboratory and field tests performed during 1985 at the existing treatment bunker. The waste characterization report resulting from these studies, titled "Laboratory and Field Testing Studies of Calcium Carbide Desulfurization Slag, April 1985," was included in the November 5, 1985, RCRA Part B Permit Application.

Laboratory testing was conducted to determine the volume of acetylene gas generated by the reaction of calcium carbide desulfurization slag with water. The purpose of these studies was to determine the



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Dwn. by:	EAP
Date:	9-9-87
Proj.#	1125.08

FIGURE 1

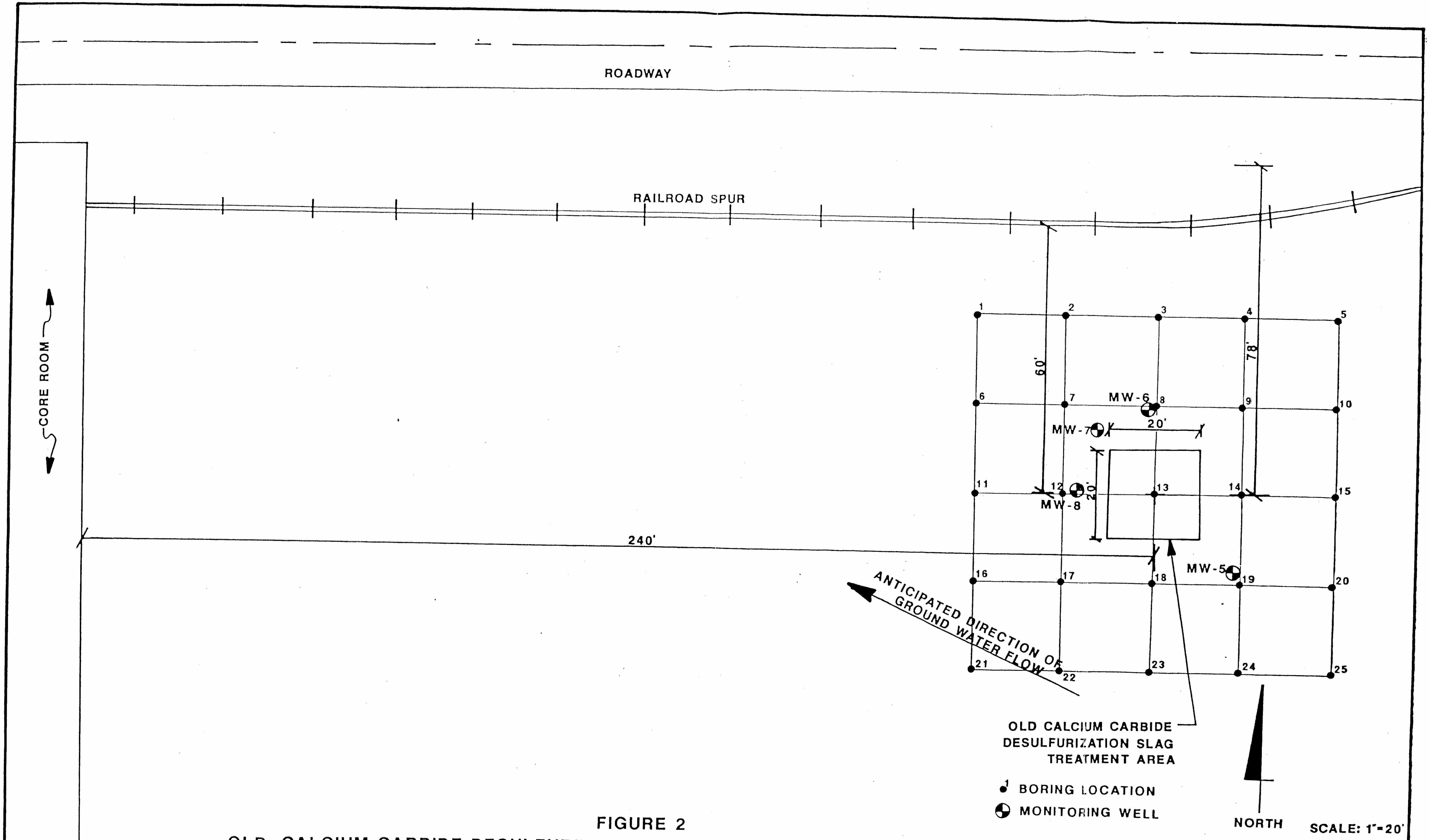


FIGURE 2
OLD CALCIUM CARBIDE DESULFURIZATION SLAG BUNKER TREATMENT CLOSURE PLAN
GM-CFD NODULAR IRON PLANT SAGINAW, MICHIGAN

RMT INC

Dwn. by: MCS
Date: 9/87
Proj.#: 1125.09

effectiveness of reacting the slag with water by releasing the product gases under various types of controlled laboratory conditions designed to simulate treatment. The experimental procedure developed by RMT for measuring acetylene gas generation capacity of desulfurization slag is based on the principle that gas displaces its own volume in water. The procedure used for this gas generation capacity test conducted in 1984-85 has been slightly revised. Refer to Appendix B of this Closure Plan for the current procedure.

The results of these simulation tests showed that water can be used to render calcium carbide desulfurization slag nonreactive (with respect to acetylene gas generation capacity) with no stirring required at a solid-to-liquid ratio as high as 1:2. The gas generating capacity of the slag was reduced from about 10 ml gas per gram of slag to 0.1 ml/gram.

The gas generating capacity of calcium carbide desulfurization slag was found to vary as a function of the solid-to-liquid ratio employed during treatment. This variance is related to the solubility of the generated gases in water. Reaction temperature was found to have no effect on the volume of gas generated. The gas generation test results are given in Table 3-1.

The treatment of calcium carbide desulfurization slag to render it nonreactive, and therefore nonhazardous, results in a mixture of desulfurization slag residue (solid) and treatment process wastewater (liquid).

For the solid residue, EP Toxicity tests were conducted on the treated CaC_2 desulfurization slag from the existing treatment bunker.

TABLE 3-1

GAS GENERATION TEST RESULTS

Date	Sample Number	Solid-To-Liquid Ratio	Stirred or Unstirred	Gas Saturated with Acetylene	Gas Generation (ml Dry Gas/ Gram Slag)
11/12/84	8349	1:4	S	No	2.41
11/12/84	8349	1:20	S	No	0.66
11/14/84	8349	1:1	S	No	9.64
11/12/84	8349	1:1	S	No	8.67
12/10/84	8349	1:20	S	Yes	8.39
01/10/85	8348	1:20	S	Yes	1.23
01/10/85	8348	1:1	S	Yes	< 0.1
01/10/85	Treated Residue	1:1	S	Yes	< 0.1

NOTE: Samples 8348 and 8349 are untreated.

Table 3-2 summarizes the results which show that the treated CaC_2 desulfurization slag residue is nonhazardous according to the characteristic of EP Toxicity.

For treatment process wastewater, the ASTM water leaching procedure was used to generate a simulated slag treatment leachate. Table 3-3 summarizes the results of a typical leachate, and indicates that the leachate would not be classified as a hazardous waste.

TABLE 3-2

EP TOXICITY TEST RESULTS FOR CALCIUM CARBIDE DESULFURIZATION SLAG AT EXISTING TREATMENT BUNKER

Parameter	MDNR Hazardous Waste Limits (mg/l)	Treated Calcium Carbide Desulfurization Slag Sample #4744 Taken 11/10/83 and 11/11/83	Treated Calcium Carbide Desulfurization Slag From Treatment Slab Sample #7548 Taken 6/28/84	Treated Calcium Carbide Desulfurization Slag From Treatment Slab Sample #7549 Taken 6/29/84
Arsenic	5.0	0.66	0.153	0.031
Barium	100.0	< 0.2	< 0.2	< 0.2
Cadmium	1.0	< 0.01	< 0.01	< 0.01
Chromium (Total)	5.0	0.08	0.10	0.07
Lead	5.0	< 0.1	< 0.1	< 0.1
Mercury	0.2	< 0.0002	< 0.0002	< 0.0002
Selenium	1.0	0.026	0.014	0.033
Silver	5.0	< 0.02	< 0.02	< 0.02
Copper	100.0	< 0.02	< 0.02	< 0.02
Cyanide	20.0	--	< 0.02	< 0.02
Zinc	500.0	0.03	< 0.02	0.02

4. PRELIMINARY SITE EVALUATION FOR OLD CALCIUM CARBIDE DESULFURIZATION SLAG TREATMENT UNIT

The objectives of conducting the Preliminary Site Evaluation are as follows:

- To identify areas of potential environmental effects near the old calcium carbide desulfurization slag treatment unit.
- To obtain basic soil and ground water data in the area being investigated.
- To obtain background soil data in areas which have not been affected by the calcium carbide desulfurization slag treatment process, but are representative of the general, industrial area.

To accomplish these objectives, a Preliminary Site Evaluation Plan was developed. Portions of the evaluation have already been completed. The evaluation includes the following:

- Four soil borings were located around the old desulfurization slag treatment unit; two of these were developed as ground water monitoring wells.
- Four soil borings were located in background areas.
- The soil boring locations (other than background) were selected in an area that would likely be affected if releases from the treatment unit had occurred.
- The ground water monitoring well locations were selected in order to position the wells near the anticipated downgradient edge of the treatment unit.
- Soil samples were collected from three depths in each boring in order to preliminarily assess the vertical extent of contamination. However, only the near-surface and mid-depth samples are being analyzed at this time. The bottom samples will be retained for possible analysis at a future date, pending the results of the upper-level samples.
- Sample collection, preservation, transport, and analysis followed USEPA-approved methods.
- Soil data analysis will consist of comparing treatment area concentrations with background concentrations and with published "natural" soil concentrations.

- Ground water data analysis will consist of comparing ground water concentrations to regulatory standards or recommended levels.

4.1 Soil Boring and Monitoring Well Locations

Four soil borings were located around the old 20' x 20' desulfurization slag treatment unit to help determine if contaminants had migrated from the area. The four locations for this preliminary investigation were selected so as to encompass the treatment area, as shown on Figure 2. The approximate locations of the four borings are summarized as follows:

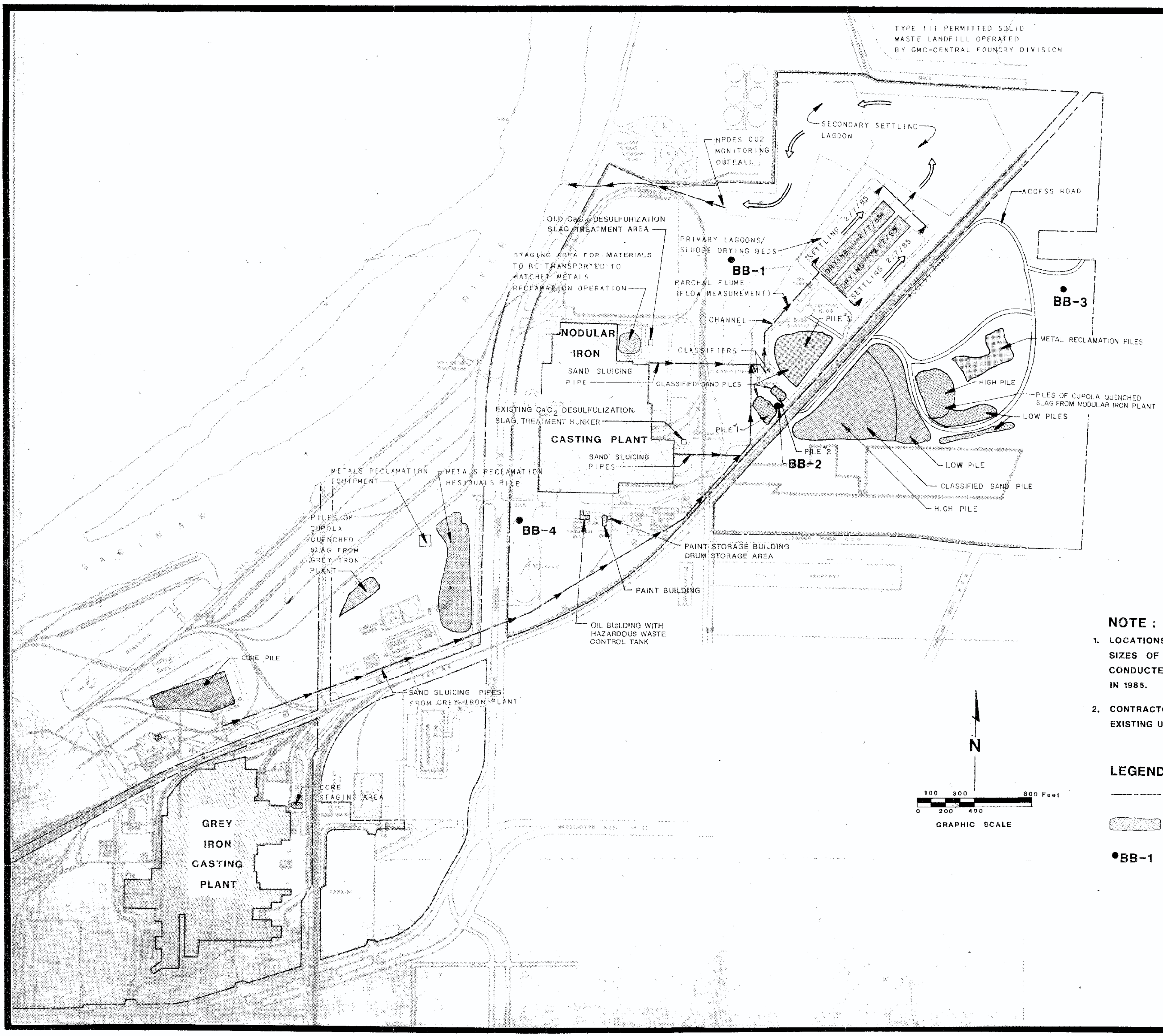
- Twenty feet northeast of the treatment area (Boring 9).
- Twenty feet northwest of the treatment area (Boring 7).
- Twenty feet southeast of the treatment area (Boring 19).
- Twenty feet southwest of the treatment area (Boring 17).

Ground water is assumed to flow toward the northwest. The two borings to the west were developed into ground water monitoring wells.

4.2 Background Areas

Four soil borings were located in areas believed to be representative of areas unaffected by desulfurization slag handling, treatment, storage, or disposal. The approximate locations for the four borings are presented in Figure 3. Analytical results of soil samples from these background borings will be used to establish a range of background concentrations for the specific parameters being analyzed.

None of the background borings were developed for ground water monitoring purposes.



NOTE :

1. LOCATIONS, DESCRIPTIONS AND APPROXIMATE SIZES OF PILES ARE BASED ON AN INVENTORY CONDUCTED BY RMT AND CFD STAFF MEMBERS IN 1985.
2. CONTRACTOR SHALL FIELD LOCATE AND VERIFY EXISTING UTILITIES PRIOR TO BORING INSTALLATION.

LEGEND

-------	APPROXIMATE CFD PROPERTY LINE
▨	STOCKPILE
●BB-1	BACKGROUND BORING LOCATION AND NUMBER

NO.	BY	DATE	REVISION	APP'D

PROJECT: OLD CALCIUM CARBIDE DESULFURIZATION
SLAG TREATMENT AREA CLOSURE PLAN
GM-CFD SAGINAW, MICHIGAN

SHEET TITLE:
BACKGROUND SAMPLE LOCATIONS

DRAWN BY EAP	SCALE	PROJ. NO. 1125.08
CHECKED BY RCK		DRWG. NO.
APPROVED BY TJJ	DATE PRINTED: DEC 21 1987	SHEET OF FIGURE 3
DATE 9-9-87		

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4.3 Soil Boring Methods

Soil borings were advanced using a hollow-stem auger, and soil samples were collected continuously with a split-spoon sampler, as described in ASTM D1586-84. At the old treatment unit, two soil borings were advanced to an 8-foot depth and two to a 15-foot depth. The deeper borings were used to install ground water monitoring wells. Four additional borings have been completed in background areas as previously described.

When sampling within a boring was completed, the borehole was backfilled with a mixture of borehole sand and bentonite grout. For the two monitoring well borings, cuttings remained on the ground surface near the boreholes and wells were installed as described in Section 4.6. Sample chain-of-custody and boring log forms (Figures 4 and 5) were completed for each boring.

In order to reduce cross-contamination during sample collection, the following cleaning procedures were used prior to starting and between individual borings:

- All augers, drill rods, and other tools and drilling equipment used in sampling were cleaned using steam-cleaner with potable water prior to use at each boring ocation.
- While on-site, none of the augers or other down-hole equipment were allowed to come into contact with surrounding soils prior to use.

4.4 Soil Sample Collection, Preservation, and Transport

In order to determine the effects of desulfurization slag treatment on different soil horizons, three soil samples were collected in each boring. Locations were based on the following schedule:

- A near-surface sample was collected within the top 24 inches of soil. In paved areas, the sample was taken from the top 24 inches of the material directly beneath the pavement.

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SAMPLE RECORD FORM

1. CLIENT:
2. JOB NUMBER:
3. LOCATION:
4. TYPE OF SAMPLES: (WATER, WASTE WATER, SOLID WASTE)
5. SAMPLING DATE:
6. SAMPLE COLLECTOR:
7. AFFILIATION:
8. SIGNATURE:
9. PURCHASE ORDER #:
10. COMMENTS

SAMPLE DESCRIPTION	RMT LABORATORY NUMBER	SAMPLE LOCATION AND DATE			
		(R,	LS,	PS,	D)*

*R (REFRIGERATOR) LS (LABORATORY STORAGE) PS (PERMANENT STORAGE) D (DESTROYED)

SUBSEQUENT ANALYSIS: SAMPLE TEMP. ACCEPTABLE: SAMPLE PRESERVED PROPERLY

SAMPLES TRANSPORTED BY:

NAME: DATE AND TIME:

AFFILIATION: SIGNATURE:

SAMPLES RECEIVED BY:

NAME: DATE AND TIME:

AFFILIATION: SIGNATURE:

SAMPLE DESTRUCTION AUTHORIZATION:

SAMPLE NUMBERS:

PROJECT MANAGER: DATE:

RMT PRINCIPAL: DATE:

FIGURE 4

RMT, Inc.
F-204 (R2-81)

LOG OF TEST BORING
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PROJECT:
LOCATION:
DRILLED BY:
LOGGED BY:
DATE:

JOB NO.:
BORING NO.:
SURFACE ELEV.:
SHEET NO.:

SAMPLE						VISUAL CLASSIFICATION and Remarks
		Recovery	Moisture			
No.	Type			N	Depth	
					5	
					10	
					15	
					20	
					25	
					30	
					35	
					40	

GENERAL NOTES
START:
COMPLETE:
RIG:
CREW CHIEF:
DRILLING METHOD:

WATER LEVEL OBSERVATIONS
WHILE DRILLING:
UPON COMPLETION:
TIME AFTER DRILLING:
DEPTH OF WATER:
DEPTH OF CAVE-IN:

FIGURE 5

- A mid-depth sample was collected 1-2 feet above the water table or the foundry sand/native soil interface (whichever occurred first).
- A deep sample was collected approximately 1-2 feet below the water table or the foundry sand/native soil interface (whichever occurred first).

Soil samples were collected continuously because the depths to ground water and to the foundry sand/native soil interface were unknown. However, only those samples at the three depths specified above were transported to the lab. Continuous sampling occurred in the upper 8 feet (approximately) of each of the eight borings (four background and four treatment area). Sampling did not continue past the 8-foot depth, even in the two borings which received monitoring wells.

After the split-spoon sampler had been removed from the center of the hollow-stem augers, the sampler was opened and a record of visual observations were made on the boring logs. Split-spoon samplers were cleaned on site using a steam-cleaner. The liquids generated from this cleaning procedure were discharged directly onto the ground near the boring locations.

For each soil sample, approximately 800 grams of soil were collected for testing purposes. The following procedures were used to prepare the sample for transport back to the RMT Laboratory:

- The sample remained in the split-spoon sampler until all split-spoons were collected from a boring.
- The split-spoons were opened, and foreign matter was trimmed from the sample.
- A subsample of approximately 800 grams was taken and placed in a one-pint glass jar provided by the laboratory. The subsample is being used for soil compositional analysis.

- A chain-of-custody form was completed and accompanied the sample during transport.

4.5 Soil Sample Selection and Analysis

Three samples were collected from each of the borings, as described in Section 4.4, and transported to the laboratory.

The near-surface sample and the mid-depth sample are being analyzed for the parameters listed in Table 4-1. Arsenic and selenium were selected for analysis because testing at the GMC-CFD Saginaw Nodular Iron Plant and at other nodular iron foundries has shown that these parameters may be present in desulfurization slag leachate. Lead and cadmium are included because they are often found in significant concentrations in ferrous foundry wastes. Concentrations of lead and cadmium in desulfurization slag are generally low. All analyses are compositional (not leaching), based on MDNR guidance.

The bottom sample from each boring will not be analyzed at this time. However, select samples may be analyzed in the future, depending on the results of the upper level analyses.

4.6 Ground Water Monitoring Well Installation Methods

Two borings were advanced to an approximate depth of 15 feet below the ground surface. Monitoring wells were installed in these borings. The wells were constructed of 4-inch PVC. All pipe and screens were factory cleaned and delivered to the site in individually wrapped containers for direct installation.

Well screens were 10 feet long and situated so the ground water surface was intercepted. Sand (washed silica or equivalent) was

TABLE 4-1

SOIL COMPOSITIONAL ANALYSIS

Parameter	Analytical Method[1]
Arsenic	7060
Cadmium	7130
Lead	7420
Selenium	7740

---

[1] "Test Methods for Evaluating Solid Waste;" USEPA (530) SW-846, November 1986, 3rd Edition.

backfilled around the screen and extended approximately two feet above the top of the well screen. A bentonite seal was installed above the sand layer. The remaining borehole annulus was filled with Portland cement concrete to seal the borehole and anchor the protective casing. The 6-inch square steel protective casings were the standard, aboveground type.

Equipment decontamination procedures have been described in Section 4.3.

4.7 Ground Water Sample Collection, Preservation, and Transport

One round of ground water samples were collected from these monitoring wells. Samples were collected within one week of installing the wells. The ground water levels were measured prior to collecting the samples.

The ground water sampling procedures are summarized as follows:

- All wells were purged by bailing to remove three to five volumes of water prior to sampling.
- The samples were collected using a PVC bailer.
- Samples or sample portions requiring filtration were filtered in the field through a 0.45 micron millipore filter.
- Samples for chemical analysis were placed in appropriate polyethylene or glass containers with proper preservatives.
- The equipment used to purge the wells and collect the samples was cleaned between each well to prevent cross-contamination. The cleaning procedure included triple rinsing with deionized water.
- A trip blank accompanied the sample containers during transport.
- Field notes and measurements were taken for water elevations and pH.

- The samples were iced and transported to the laboratory under Chain-of-Custody procedures.
- The samples will be analyzed according to USEPA-approved procedures and strict QA/QC protocols.

4.8 Ground Water Analysis

Two ground water samples were collected from the monitoring wells placed near the treatment area. The analytical parameters and method references are presented in Table 4-2.

4.9 Preliminary Site Evaluation Report

A brief report will be prepared following the completion of the on-site activities and sample analyses described in Section 4 of this Closure Plan. The report will include the following:

- Summary of the on-site activities.
- Laboratory results.
- Comparisons of soil concentrations from the old treatment unit with concentrations from background areas and with "natural" soil concentrations from the literature.
- Comparisons of ground water concentrations with regulatory standards and recommended levels.
- Recommendations for further sampling and testing if needed.

TABLE 4-2

GROUND WATER ANALYSIS FOR EXISTING TREATMENT BUNKER

Parameter	Analytical Method[1]	Preservation[2]	Volume (ml)
Arsenic	206.2	Nitric	50
Cadmium	213.2, 200.7	Nitric	50
Chromium (Total)	218.2, 200.7	Nitric	50
Lead	239.2	Nitric	50
Selenium	270.3	Nitric	50
Alkalinity	310.1	None	100
Chloride	325.2	None	100
Fluoride	340.2	None	100
pH	150.1	None	100
Total Phenols	420.2	Sulfuric (Glass)	500
Total Organic Carbon	415.2	Sulfuric (Glass)	100

[1] "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-84-017.

[2] Samples in plastic containers unless otherwise noted.

5. CLOSURE PERFORMANCE STANDARD

GMC-CFD Saginaw Nodular Iron must close the old calcium carbide desulfurization slag treatment unit in a manner that satisfies 40 CFR 265.111. To accomplish this, the regulations indicate that GMC-CFD Saginaw Nodular Iron must:

- minimize the need for further maintenance; and
- control, minimize or eliminate post-closure escape of hazardous waste or hazardous constituents to the environment.

These requirements will be satisfied by documenting that:

- all waste residues are treated and removed;
- the concrete slab of the treatment unit is decontaminated; and
- surrounding soils have not been adversely affected by release of hazardous constituents from the desulfurization slag.

As specified in 40 CFR 265.258, characteristically hazardous items must be managed as hazardous wastes unless the waste no longer exhibits the characteristics of a hazardous waste.

As described in the introduction, calcium carbide desulfurization slag is classified as a hazardous waste by virtue of the characteristic of reactivity. Since treatment with water renders the desulfurization slag nonhazardous, no provisions for post-closure activities are included.

The closure performance standard will be removal of all treated desulfurization slag and other affected soils, and decontamination of associated equipment used to implement this plan.

The following is a step-by-step description of how the GMC-CFD Saginaw Nodular Iron Plant will close the old calcium carbide desulfurization slag treatment unit (these steps will be observed by representatives of both the Saginaw Nodular Iron Plant, as the owner/operator, and an independent registered professional engineer). This work will follow the preliminary site evaluation activities described in Section 4, and is based on attaining "clean closure."

1. The sampling and analysis plan, detailed in Section 6 of this Closure Plan, will be implemented.

2. If the results of the sampling and analysis plan show that the soils in the vicinity of the treatment unit have not been adversely impacted, then closure will be considered complete, and documentation as described below will be provided.

3. If adverse environmental impact is determined for soil in the vicinity of the unit, that material will be removed and disposed at a suitable off-site landfill. Additional samples will then be collected until underlying and adjacent material indicates no direct adverse environmental impact attributed to the desulfurization slag treatment operation. Michigan DNR procedures dated April 30, 1987, as prepared by the Hydrogeologic Review Unit, will be the basis for statistical analysis and evaluation.

4. Ground water monitoring (Section 7) will be conducted only if significant contamination of native soil is found.

5. A closure documentation report will be submitted to the MDNR for approval after closure has been completed. The documentation of closure shall be performed by both Saginaw Nodular Iron and an independent professional engineer, registered in the State of Michigan. The report will include chain-of-custody and laboratory analytical reports for all samples collected and analyzed.

6. SAMPLING AND ANALYSIS PLAN

Preface

This sampling and analysis plan has been developed for use in documenting "clean closure" of the treatment unit. The results of the Preliminary Site Evaluation may show this approach to be inappropriate. The decision process described in Section 2 (Diagram 1) will be used to decide if and when to implement this part of the plan.

6.1 Approach

Samples in the immediate area of the treatment unit will be collected and analyzed in accordance with MDNR guidelines for grid spacing, testing, and chemical parameters as specified in the MDNR's May 5, 1987, memorandum entitled "How Clean in Clean II." For comparison purposes, background samples will be collected from areas that have not been affected by desulfurization slag treatment, handling, or storage. Pending results of the Preliminary Site Evaluation, additional background soil samples may be collected and analyzed prior to implementing this Sampling and Analysis Plan (Diagram 1). The concentrations from samples collected near the old treatment unit will then be statistically compared to concentrations from samples collected in background areas. This comparison will determine if operation of the desulfurization slag treatment unit has affected the adjacent soil.

The Sampling and Analysis Plan calls for borings in twenty-five locations on a grid surrounding the treatment unit and at four background locations. Four of the 25 borings have already been completed as part of the Preliminary Evaluation. The borings will penetrate the overlying fill materials and extend to the underlying native soil. Samples will be collected at the surface, at the foundry sand/native soil interface, and two feet into the native soil.

Water leach tests as well as compositional (total) analysis will be performed on the soil samples. Laboratory methods will consist of EPA-approved analytical techniques, except in cases where there is no EPA-approved analytical technique. In those cases, a proposed analytical technique is included in this plan.

6.2 Boring Locations

A soil sampling grid system has been established according to guidelines specified by the MDNR in their memorandum of May 5, 1987, entitled "How Clean is Clean II," and USEPA Document SW 846. The MDNR specified that a grid system be established that would cover the treatment area as well as 30 feet from the unit in all directions. The grid interval of 20 feet was determined to be appropriate according to the provisions of Part B "Sampling Grid" of the above-referenced MDNR memorandum. Each grid section will be 20 foot by 20 foot as shown in Figure 2. This results in a 5 x 5 grid with a total of 25 borings. The suggested locations of the borings are presented in Figure 2. Locations of these borings may change slightly to avoid existing utilities in the area of the sampling grid.

In addition to the four borings in the vicinity of the treatment unit, four borings have been placed in areas of similar soils, located some distance from the waste treatment unit and not affected by desulfurization slag handling, storage or treatment. This was done as part of the Preliminary Site Evaluation. Samples from these four borings are being used to establish the range of background concentrations for the specified parameters. However, because this Sampling and Analysis Plan calls for more chemical parameters than the Preliminary Site Evaluation did, four additional background borings will need to be drilled and sampled. The approximate locations of these four borings are indicated in Figure 3.

6.3 Sample Collection, Preservation, and Shipment

Three samples will be collected from each boring using a split-spoon sampler through hollow-stem augers as described in ASTM D1586-84. The recently conducted Preliminary Site Evaluation activities indicate that the ground water table is approximately 8 feet below the ground surface. On the basis of this information, we anticipate that split-spoon samplers will be continuously collected to a depth of ten feet or less. Soil samples will be collected near the ground surface and at the top of the water table. A sample will also be collected 1 to 2 feet below the foundry sand/native soil interface.

In order to reduce cross-contamination during sample collection, the driller will use cleaning procedures prior to starting and between individual borings which are similar to the following:

- All augers, drill rods, and other tools and drilling equipment used in sampling will be steam-cleaned or washed with soapy water and rinsed with water from the City of Saginaw's public water supply system prior to use at each boring location.
- To avoid equipment contamination, all cleaning will be done at a site not used for desulfurization slag treatment, storage, or handling.

- While on site, none of the augers or other down-hole equipment will be allowed to come into contact with surrounding soils prior to use.

Split-spoon samplers will be cleaned prior to starting and between individual borings using the following procedures (or equivalent):

- Split-spoon samplers will be steam-cleaned or washed with soapy (laboratory-grade detergent) water and scrubbed with a wire brush to remove soil.
- After being washed with soapy water, the split-spoon sampler will be rinsed with potable water.
- If oily soil is encountered, the samplers will be rinsed with hexane, methanol, or similar organic solvent.
- The split-spoon samplers will receive a final rinse with distilled water.

Liquid used in cleaning will be discharged directly onto the ground. After the split-spoon sampler has been removed from the center of the hollow-stem augers, the sampler will be opened and visual observations will be recorded on the boring log.

For each soil sample, approximately 2,600 grams of soil will be required to conduct the compositional analyses and leaching tests. Because of the large amount of sample required to run these analyses, an over-sized split-spoon sampler (3" inside diameter) will be used. The following procedures will be used to prepare the sample for transport back to the laboratory:

- Foreign matter will be trimmed from the split-spoon sample.
- The sample will be placed in a plastic jar and homogenized by mixing. Exposure to the atmosphere should be minimized during sample preparation.

- A subsample for carbide reactivity will be taken from the plastic jar and placed in a one-pint metal can.
- The remaining sample will be used for soil compositional analysis, and transported to the lab using chain-of-custody forms.

When a boring is completed, it will be backfilled with a cement/bentonite grout. Boring logs will be completed for all boreholes. A chain-of-custody form will accompany all samples back to the laboratory. Quality assurance/quality control measures for sample collection and handling are described in Appendix C. If GMC-CFD Saginaw Nodular Iron elects to use another lab, equivalent QA/QC data will be submitted for approval by the MDNR.

6.4 Sample Analyses

Upon arrival at the laboratory, the soil samples will be analyzed for the parameters listed in Table 6-1. All analyses will be performed according to EPA-approved procedures except in cases where there are no approved methods for analysis. Soil compositional analysis will be performed according to USEPA Document SW 846. Leachate will be generated using ASTM Method D3987, and analyzed according to EPA 600/4-84-017. Table 6-1 includes the applicable method reference number for each parameter.

There are no EPA-approved methods for analyzing total organic carbon, fluoride or total phenols in soils, or for analyzing calcium carbide in soils or water. For this reason, RMT proposes that these parameters be measured in the following way:

- Total organic carbon. Since inorganic carbon cannot be removed from a solid sample, a TOC instrument will actually

TABLE 6-1

PARAMETERS FOR SOIL COMPOSITIONAL ANALYSIS AND ASTM WATER LEACHATE TESTS

Parameter	Soil Compositional Method[1]	ASTM Water Leachate Method[2]
Arsenic	7060	206.2
Cadmium	7130	213.2, 200.7
Chromium	7190	218.2, 200.7
Iron	7380	236.1
Lead	7420	239.2
Selenium	7740	270.3
Zinc	7950	289.1
Total Organic Carbon	--	415.2
Total Phenols (4AAP)	--	420.2
Fluorides	--	340.2
Calcium Carbide	--	--

[1] Approved method according to the 3rd Edition of USEPA document SW 846 "Test Methods for Evaluating Solid Wastes, November 1986."

[2] Approved method according to the "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, EPA 600/4-82-055, and EPA 600/4-84-017.

measure total carbon. Total carbon will be determined by high temperature combustion of all carbon to carbon dioxide, followed by measurements of the carbon dioxide evolved.

- Fluoride. The sample will be crushed to render it homogeneous, then fused with sodium carbonate at a high temperature to form sodium fluoride. The fusion product will be dissolved in acid. Fluoride will be measured by colorimetric or specific ion method depending on concentration.

- Total phenols. A 1-10 g sample will be mixed with distilled water. The mixture will then be subjected to the distillation step from USEPA 600/4-79-020 Method 420.1 for water and waste. Total phenols will be measured in the distillate.

- Calcium carbide. The calcium carbide content of soils will be measured indirectly using the method presented in Appendix D.

  Any calcium carbide contained in a soil sample subjected to the ASTM water leach test will be reacted. Consequently, calcium carbide will be deleted as a parameter for the ASTM water leachate analysis.

6.5 Statistical Comparison

In order to determine if there has been an increase in the concentrations of specified parameters in the soils surrounding the old (original) desulfurization slag treatment unit, a statistical analysis of the data will be used. This analysis will be performed in accordance with the methods presented in 40 CFR 264 Appendix IV except that the Cochran's approximation to the Student t-test will be used to establish the upper confidence limit at the 99% confidence level for background concentrations. This analysis will be performed for the data generated from the soil compositional analyses and ASTM water leachate analyses. To determine if the old treatment unit has impacted the surrounding soils, the analytical results of the individual soil samples collected from the area of the desulfurization slag treatment unit will be compared to the upper confidence limit for the background soil sample results. This comparison will be done on a parameter-by-parameter basis.

7. GROUND WATER MONITORING

One round of ground water samples is being analyzed as part of the Preliminary Site Evaluation. If the results of the soil sampling and analysis plan (Section 6) indicate that significant contamination of native soils (due soley to the treatment of desulfurization slag) has not occurred, then additional ground water monitoring will not be performed and GMC-CFD will initiate preparation of the Closure Documentation Report. If significant soil contamination has been shown to result from the treatment operations, then 2 additional shallow wells, one upgradient and one downgradient, will be installed for the purpose of conducting ground water monitoring in the vicinity of the old treatment unit.

Samples from the four monitoring wells at the old treatment unit will be collected once a month for four consecutive months and analyzed for the parameters shown in Table 7-1.

If the results of the ground water monitoring program, in combination with the results obtained from the soil sampling program, indicate that the native soil has not been contaminated, then the above program shall constitute completion of ground water monitoring in support of clean closure for the treatment unit and vicinity.

If the soil and ground water results indicate contamination of both the soils and shallow ground water, then GMC-CFD Saginaw Nodular Iron will install four cluster wells. These wells will be installed to evaluate the aquifer(s) beneath the unit. After installation of these wells, another four months of deep well ground water samples will be collected and analyzed according to the statistical methods described in Section 6.5 of this Closure Plan.

TABLE 7-1

GROUND WATER MONITORING PARAMETERS

Parameter	Ground Water Method[1]	Parameter	Ground Water Method[1]
Arsenic	206.2	Calcium	200.7, 215.1
Cadmium	213.2, 200.7	Chlorides	325.2
Chromium	218.2, 200.7	Fluorides	340.2
Iron	236.1	Manganese	200.7, 243.1
Lead	239.2	Nitrate	353.2
Selenium	270.3	Total Phenols	420.2
Zinc	289.1	Sodium	273.1
pH	150.1	Total Phosphorus as P	365.2
Total Alkalinity	310.1	Potassium	200.7, 258.1
Carbonate Alkalinity	310.1	Total Organic Carbon	415.2
Bicarbonate Alkalinity	310.1	Magnesium	200.7, 242.1

[1] Approved method according to the "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, EPA 600/4-82-055, and EPA 600/4-84-017.

8. HEALTH AND SAFETY PLAN

All regulations concerning health and safety shall be rigorously abided. Prior to starting the work, a site-specific Health and Safety Plan will be developed to cover workers on the site. The workers' employer will be responsible for implementing the plan, directing the training of personnel, and for providing safety equipment and incidentals as required. At a minimum, the plan will address the following:

a. Hazard Evaluation, Chemical and Physical.

b. Levels of Protection

  - Personal protective clothing
  - Respiratory protection

c. Air Monitoring

d. Site Control

  - Work zones
  - Decontamination procedures; personnel and equipment
  - Site security

e. Contingency Plan

f. Medical Surveillance and Certification

g. Worker Training Certification

The Plan will be directed at compliance with applicable federal, state, and local requirements. The following references may be used to assist in the development of the Health and Safety Plan.

A. "Standard Operating Safety Guides," USEPA, November 1984.

B. "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities," NIOSH/OSHA/USCG/EPA, October 1985.

C. U.S. Department of Labor, Occupational Safety and Health Standards and Regulations including, but not limited to, 29 CFR 1910.120 on Hazardous Waste Operations.

9. ESTIMATE OF MAXIMUM WASTE INVENTORY

The RCRA regulations for closure plans (40 CFR Part 265.112[a][2]) require that the maximum inventory of wastes in storage at anytime during the life of the facility be reported. Based on discussions with plant personnel, the maximum amount of hazardous waste that was ever in storage in the facility was no greater than 1,000 tons of untreated desulfurization slag.

At the present time, untreated desulfurization slag is not being stored at the old treatment unit. GMC-CFD does not plan to store untreated slag at this old treatment unit in the future.

10. ESTIMATED CLOSURE DATE AND CLOSURE SCHEDULE

GMC-CFD Saginaw Nodular Iron intends to perform closure activities based on the following RCRA requirements (40 CFR 265.113):

- Closure must begin 90 days after receiving the final volume of hazardous waste, or 90 days after approval of the Closure Plan, whichever is greater.
- Closure must be complete 180 days after receiving the final volume of hazardous waste, or 180 days after approval of the Closure Plan.

Preparation of the Closure Documentation Report will be completed within another 3-4 weeks. A closure schedule follows:

Closure Action	Time for Completion Following Initiation of Closure (Weeks)
1. Contract for closure work.	0-4
2. Treat and dispose all accumulated calcium carbide desulfurization slag and other cleanup residuals.	2-4
3. Decontaminate the old treatment unit.	4-6
4. Perform sampling and analysis of soils to document "clean" closure, including Preliminary Site Evaluation.	6-16
5. Excavate, treat, and/or dispose contaminated soils (if necessary).	16-24
6.* Perform additional sampling and analysis to document "clean" closure (if necessary).	22-26*
7. Prepare Draft Documentation Report.	26-30
8. Submit Final Documentation Report to MDNR.	30-32

GMC-CFD estimates that closure will begin during 1988.

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* Closure activities complete after this item.

11. CLOSURE DOCUMENTATION

When closure is complete, GMC-CFD Saginaw Nodular Iron will submit a Closure Documentation Report to the MDNR. Observation of closure activities will be performed by the GMC-CFD Environmental Coordinator and an independent professional engineer, registered in the State of Michigan. The Closure Documentation Report will document that closure has taken place according to the approved Closure Plan.

APPENDIX A

MDNR INTERIM STATUS LAND DISPOSAL CLOSURE AND POST-CLOSURE CHECKLIST

FACILITY NAME : GMC-CFD SAGINAW NODULAR IRON
EPA IDENTIFICATION NUMBER : MID 041793340
STATE IDENTIFICATION NUMBER :
(Authorized States)

DRAFT

OLD CALCIUM CARBIDE DESULFURIZATION SLAG TREATMENT UNIT

INTERIM STATUS LAND DISPOSAL CLOSURE AND POST-CLOSURE CHECKLIST

		PROVIDED (Y/N)	ADEQUATE (Y/N)	NOT APPLICABLE	COMMENTS/LOCATION IN PLAN
A.	Description of Operation	Y			SECTION 1
B.	Closure Performance Standard	Y.			SECT. 5
C.	Closure of Land Disposal Units			NA	
D.	Closure of Waste Pile	Y			SECT. 4 + 6
D1.	Removal and Decontamination	Y			SECT. 4 + 6
D1a.	Waste Residues	Y			SECT. 4 + 6
D1b.	System Components	Y			SECT. 4 + 6
D1c.	Contaminated Subsoils	Y			SECT. 4 + 6
D1d.	Contaminated Equipment and Structures	Y			SECT. 4 + 6
D2.	Confirmatory Testing	Y			SECT. 4+6
D2a.	Representative Sampling Scheme	Y			SECT. 4+6
D2b.	Number of Samples	Y			SECT. 4+6
D2c.	Depth of Samples	Y			SECT. 4+6
D2d.	Horizontal Location of Samples	Y			SECT 4 + 6

. OLD TREATMENT UNIT

		PROVIDED (Y/N)	ADEQUATE (Y/N)	NOT APPLICABLE	COMMENTS/LOCATION IN PLAN
D2e.	Sampling Method/Device	Y			SECT. 4 + 6
D2f.	Chain of Custody	Y			SECT 4 + 6
D2g.	Sample Preservation Holding Time	Y			SECT 4 + 6
D2h.	Parameters and Rationale	Y			SECT. 3, 4 + 6
D2i.	Analytical Methods	Y			SECT 4 + 6
D2j.	Removal Standard/Statistical Testing	Y			SECT 4 + 6
E.	Closure of Surface Impoundment			NA	
E1.	Closure by Removal			NA	
E1a.	Standing Liquids			NA	
E1b.	Waste and Waste Residues			NA	
E1c.	Liner			NA	
E1d.	Contaminated Soil			NA	
E2.	Confirmatory Testing			NA	
E2a.	Representative Sampling Scheme			NA	
E2b.	Number of Samples			NA	
E2c.	Depth of Samples			NA	
E2d.	Horizontal Location of Samples			NA	
E2e.	Sampling Method/Device			NA	

OLD TREATMENT UNIT

		PROVIDED (Y/N)	ADEQUATE (Y/N)	NOT APPLICABLE	COMMENTS/LOCATION IN PLAN
E2f.	Chain of Custody			NA	
E2g.	Sample Preservation/Holding Time			NA	
E2h.	Parameters and Rationale			NA	
E2i.	Analytical Methods			NA	
E2j.	Removal Standard/Statistical Testing			NA	
E3.	Non-Hazardous Demonstration			NA	
E3a.	Analytical Results			NA	
E3b.	Representative Sampling Scheme			NA	
E3c.	Depth of Samples			NA	
E3d.	Horizontal Location of Samples			NA	
E3f.	Chain of Custody			NA	
E3g.	Sample Preservation/Holding Time			NA	
E3h.	Parameters and Rationale			NA	
E3i.	Analytical Methods			NA	
E3j.	Removal Standard/Statistical Testing			NA	
E4.	Waste Remains After Closure			NA	

OLD TREATMENT UNIT

		PROVIDED (Y/N)	ADEQUATE (Y/N)	NOT APPLICABLE	COMMENTS/LOCATION IN PLAN
F.	Closure and Post-Closure	Y			SECT. 2 + 5
F1.	Objective of Closure and Post-Closure	Y			SECT 2 + 5
F1a.	Control of Pollutant Migration	Y			SECT 2, 4, 5 AND 6
F1b.	Control of Surface Water Infiltration			NA	
F1c.	Prevention of Erosion			NA	
F2.	Considerations			NA	
F2a.	Type and Amount of Hazardous Waste			NA	
F2b.	Mobility and Expected Migration Rate			NA	
F2c.	Site Location			NA	
F2d.	Climate			NA	
F2e.	Characteristics of Cover			NA	
F2f.	Geology and Hydrology of Site			NA	
F3.	Cover Design			NA	
F3a.	Layer Thickness			NA	
F3b.	Final Contours			NA	
F3c.	Grain Size Requirements			NA	

OLD TREATMENT UNIT

		PROVIDED (Y/N)	ADEQUATE (Y/N)	NOT APPLICABLE	COMMENTS/LOCATION IN PLAN
F3d.	Engineering Indices			NA	
F3e.	Compaction/Density Requirements			NA	
F3f.	Cover Vegetation			NA	
F3g.	Permeability Requirements			NA	
F3h.	Slope Stability Analysis			NA	
F3i.	Synthetic Membrane Specifications			NA	
F3j.	Frost Effects			NA	
F3k.	Settlement/Subsidence Effects			NA	
F3l.	Prevention of Erosion			NA	
F3m.	Gas Collection			NA	
F4.	Construction Procedures			NA	
F4a.	Equipment Requirements			NA	
F4b.	Lift Thickness			NA	
F4c.	Construction Quality Control			NA	
G.	Disposal or Decontamination	Y			SECT 4 + 6
H.	Certification of Closure	Y			SECT 5 + 11

OLD TREATMENT UNIT

		PROVIDED (Y/N)	ADEQUATE (Y/N)	NOT APPLICABLE	COMMENTS/LOCATION IN PLAN
I.	Schedule for Closure	Y			SECT 10
J.	Notice to Local Authority			NA	
K.	Notice in Deed			NA	
L.	Maintenance			NA	
L1a.	Final Cover			NA	
L1b.	Groundwater Monitoring System			NA	
L1c.	Leachate Collection			NA	
L1d.	Gas Collection			NA	
M.	Security			NA	
N.	Groundwater Monitoring	Y			SECT 7
N1.	Groundwater Monitoring System	Y			SECT 7
N2.	Sampling and Analysis	Y			SECT 4, 6 + 7
N3.	Preparation, Evaluation, Response	Y			SECT 4, 6 + 7
N4.	Assessment Plan	Y			SECT 4, 6 + 7

APPENDIX B

GAS GENERATION PROCEDURE USED FOR PREVIOUS TESTING

CALCIUM CARBIDE DESULFURIZATION SLAG
ACETYLENE GENERATION PROCEDURE

CAUTION: THIS TEST SHOULD ONLY BE DONE UNDER A HOOD BY TRAINED LABORATORY PERSONNEL

BACKGROUND

This test procedure describes a method that can be used to provide reproducible measurements of acetylene generation resulting from the addition of deioinized water to calcium carbide desulfurization slag. During development of the test, it was found that gas generation could be affected by several factors, including gas solubility and the rate of stirring of the reaction flask. This procedure is designed to establish conditions for the above variables so that reproducible test results are attainable. This procedure is not intended to simulate actual treatment of these wastes, but rather is primarily intended to measure the gross acetylene gas generation volume from a measured sample of slag.

The reaction of solid calcium carbide with water is shown below:

$$CaC_2 + 2H_2O = C_2H_2 + Ca(OH)_2$$

The presence of lime produces an alkaline solution (or leachate). To date, all tests have been done on an unadjusted leachate within a pH range of approximately 11.5 to 12.6.

APPARATUS REQUIRED

1. Magnetic Stirrer and Round Stirring Bar
2. Modified Erlenmeyer Gas Generation Flask
3. One-Gallon Plastic Bucket
4. 500-ml or 1000-ml Graduated Cylinder
5. 250-ml Separatory Funnel with Stopper
6. Miscellaneous Tubing, Fittings and Clamps as Shown on Drawing
7. Silicone Vacuum Grease.

REAGENTS

1. DI Water
2. Acetylene Gas

PROCEDURE

This section is divided into the following subsections, each covering a specific part of the overall test procedure:

1. Equipment Setup
2. Sample Storage
3. Sample Preparation

4A. Unreacted Sample Test
4B. Post Monitoring Effectiveness Test

Parts 1 through 3 are required for each test. Either Part 4A or 4B is required, depending on the sample.

1. Equipment Setup

1.1 The bucket should be filled with DI water and allowed to sit for several minutes to remove any entrained air.

1.2 The remainder of the equipment should be set up according to the procedure described in Part 4A or 4B.

2. Sample Storage and Preparation

2.1 All samples for testing should be collected in sealed air-tight and moisture-proof containers.

2.2 Samples should be tested within 1 week of sample collection.

3. Sample Preparation

3.1 The sample weight used for the test will be 100 grams (based on a solid-to-liquid ratio of 1:1). If any other solid-to-liquid ratio is used, the following table gives the recommended sample weight and water volume to be used (the 1:1 solid-to-liquid ratio should be used for the Post-Monitoring Effectiveness test--see Procedure 4B). Tests at other solid-to-liquid ratios would simulate solid-to-liquid ratios for open bunker treatment of these wastes.

Solid-to-Liquid Ratio of Test	Grams of Sample	Volume of Water-ml
4.00	100	25
2.00	100	50
1.00	100	100
0.50	100	200
0.25	50	200
0.10	10	100
0.05	10	200

Record weight on the data sheet.

4. Acetylene Generation Test

Note: This section is divided into two parts. Part A should be used on all samples of dry unreacted slag. Part B is applicable to all treated and moist untreated samples and is also the Post-Monitoring Effectiveness Test.

PART A

4A.1 The weighed sample is added to the Erlenmeyer Gas Generation Flask. Make certain the line leading into the inverted graduated cylinder is in the air, and clear of any water in its line.

4A.2 Add deionized water to the separatory funnel and proceed with acetylene saturation.

CAUTION: ACETYLENE GAS IN AN EXTREMELY FLAMMABLE MATERIAL. THE FOLLOWING PROCEDURE SHOULD ALWAYS BE CONDUCTED IN A LABORATORY HOOD BY TRAINED PERSONNEL.

4A.3 Saturation of the DI water is done within the separatory funnel.

4A.4 Saturation of the air headspace is done by purging acetylene gas through the inverted graduated cylinder.

4A.5 The regulator should be carefully opened until a discharge pressure of not more than 7 psig is obtained.

4A.6 The gas flow rate through the fritted tube should be carefully regulated and watched throughout the saturation process. The instructions for the gas regulator should be used if the operator has any uncertainties about the use of the regulator.

<u>CAUTION</u>: IT IS VERY IMPORTANT TO MAKE SURE THAT ONLY A LOW FLOW REGULATOR IS USED FOR THIS PROCEDURE. USING A REGULAR ACETYLENE GAS REGULATOR COULD LEAD TO PERSONAL INJURY SINCE THE LINE FROM THE TANK TO THE FRITTED BUBBLER CANNOT WITHSTAND HIGH INLET PRESSURES.

4A.7 Ten to fifteen minutes should be sufficient for saturating the water in the reaction flask and the system's air headspace.

4A.8 Discontinue saturation and cover the separatory funnel with the stopper.

4A.9 Read the volume of any trapped air in the inverted graduated cylinder and record it on the data sheet.

4A.10 Make certain that the magnetic stirrer is on and functioning properly before adding any of the acetylene saturated water to the sample.

4A.11 Read and record the barometric pressure in the laboratory and record it on the data sheet.

4A.12 Measure and record the water temperature in the reservoir on the data sheet.

4A.13 To start the reaction, turn the stopcock of the separatory funnel until a constant drip is obtained. Check the flask periodically to ensure that the gas being generated is collected in the inverted graduated cylinder. The rate at which water is added to the reaction flask should be controlled to the extent that sudden releases of gas are prevented, thus protecting the integrity of the gas-tight connections.

4A.12 Measure and record on the data sheet the actual volume of water in the inverted cylinder to the same water level as the bucket. <u>This measurement should be taken 60 minutes after the start of the test</u>.

4A.13 Calibrate the pH meter according to the proper procedure.

4A.14 Measure and record the pH of the reaction flask solution (leachate) on the data sheet.

PART B

4B.1 The weighed sample is added to the reaction flask and should be distributed around the outer circumference of the flask bottom with a long spatula or similar device. The round magnetic stirring bar should be carefully placed in the center of the flask away from the slag sample. The sample weight should be recorded on the data sheet.

CAUTION: ACETYLENE GAS IS AN EXTREMELY FLAMMABLE MATERIAL. THE FOLLOWING PROCEDURE SHOULD ALWAYS BE CONDUCTED IN A LABORATORY HOOD BY TRAINED PERSONNEL.

4B.2 Saturation of the DI water is done in a large beaker, not in the reaction flask.

4B.3 The regulator should be carefully opened until a discharge pressure of not more than 7 psig is obtained.

4B.4 The gas flow rate through the fritted tube into the beaker should be carefully regulated and watched throughout the saturation process. The instructions for the gas regulator should be used if the operator has any uncertainties about the use of the regulator.

CAUTION: IT IS VERY IMPORTANT TO MAKE CERTAIN THAT ONLY A LOW FLOW REGULATOR BE USED FOR THIS PROCEDURE. USE OF A REGULAR ACETYLENE GAS REGULATOR COULD LEAD TO PERSONAL INJURY SINCE THE LINE FROM THE TANK TO THE FRITTED BUBBLER CANNOT WITHSTAND HIGH INLET PRESSURES.

4B.5 Ten to fifteen minutes should be sufficient for saturating the water in the beaker.

4B.6 The acetylene-saturated water should be placed in the separatory funnel. The water volume should be entered on the data sheet. Be absolutely sure that the stopcock on the separatory funnel is closed before the water is added.

4B.7 Read the volume of any trapped air in the inverted graduated cylinder and record it on the data sheet.

4B.8 Read and record the barometric pressure in the laboratory and record it on the data sheet.

4B.9 Measure and record the temperature of the water in the reservoir on the data sheet.

4B.10 To start the test, the magnetic stirrer should be turned on and rotating freely within the flask. The stopcock from the separatory funnel should be opened and all the water added to the reaction flask. At this point the magnetic stirrer speed should be increased to make certain that all of the slag is completely wetted and submerged within the volume occupied by the water.

4B.11 Continue stirring the slag-water mixture for one hour.

4B.12 Measure and record the actual volume of collected gas in the graduated cylinder on the data sheet.

4B.13 Calibrate the pH meter according to proper procedure.

4B.14 Measure and record the pH of the reaction flask solution (leachate) on the data sheet.

LIMS TEST ID: CMP-CARB

	Initials	Date
Calculations Checked		
QC Checked		
LIMS In		
LIMS Entry Checked		

TEST: CALCIUM CARBIDE DESULFURIZATION SLAG ACETYLENE GENERATION

CLIENT: ______________________ DATE: ______________

JOB NUMBER: __________________ ANALYST: ______________

SAMPLE NUMBER: __________ SAMPLE DESCRIPTION: ______________

TEST RESULTS

1. Sample Weight (g): ______________
2. Water Volume (ml): ______________
3. Solid-to-Liquid Ratio (#1/#2): ______________
4. Barometric Pressure (mm of Hg): ______________
5. Vapor Pressure of Water (mm of Hg): ______________
6. Reservoir Water Temperature (deg C): ______________
7. Corrected Pressure (#4-#5) mm of Hg: ______________
8. Pressure Ratio at STP (#7/760) mm of Hg: ______________
9. Starting Volume in Graduated Cylinder (ml): ______________
10. Final Volume in Graduated Cylinder (ml): ______________
11. Actual Volume of Gas (#10-#9) ml: ______________
12. Temperature Ratio at STP ((273.2)/(273.2+#6): ______________
13. Gas Volume at STP-ml (#11 X #12 X #8): ______________
14. Gas Volume at STP-ml/100 g slag (#13/#1 X 100): ______________
15. Blank Gas Volume at STP - ml/100 ml DI Water: ______________
16. Final Gas Volume at STP - ml/100 g (#14-#15): ______________
17. pH of Reaction Flask Solution (standard units): ______________

COMMENTS: ______________________________

APPENDIX C

EMT LABORATORY QUALITY CONTROL PROGRAM

RMT, Inc.
Suite 124
1406 East Washington Ave.
Madison, WI 53703-3009
Phone: 608-255-2134

RMT, INC. LABORATORY QUALITY CONTROL PROGRAM

Prepared by:

Kenneth C. Brunner
Acting Laboratory Director

Signed: Kenneth C Brunner

Asst. Laboratory Director

Signed: ______________________

LF-289 (R11/87)

RMT, INC. LABORATORY
QUALITY CONTROL PROGRAM

I. PERSONNEL

STAFF MEMBER	TITLE	ACADEMIC
KENNETH C. BRUNNER	LABORATORY DIRECTOR, ACTING	M.S.
OPEN	ASST. LAB DIRECTOR	
SUSAN E. WELLS	ADMINISTRATIVE SUPERVISOR	B.S.
ERIC L. THOMAS	INORGANICS SUPERVISOR	B.S.
OPEN	ORGANICS SUPERVISOR	
KARLA HALL	LEAD WORKER - METALS	B.S.
OPEN	LEAD WORKER - ORGANICS	
SYED ALAM	LABORATORY ANALYST	M.S.
KATHLEEN ARNOLD	LABORATORY AIDE	B.S.
JOE CEBE	LABORATORY AIDE	A.D.
MARIJANE CURRY	LABORATORY ANALYST	B.S.
LAURIE DUNN	LABORATORY AIDE	M.S.
MARCIA ECKMEYER	LABORATORY AIDE	HIGH SCHOOL
ROSANNE GATES	SAMPLE ENTRY TECHNICIAN	HIGH SCHOOL
JEFF GEARHART	LABORATORY ANALYST	B.S.
JULIE HECKL	LABORATORY ANALYST	WORKING ON B.S.
SHARON KOCH	ADMINISTRATIVE ASSISTANT	HIGH SCHOOL
PAUL KORGER	LABORATORY ANALYST	B.S.
MICHAEL MILLER	LABORATORY ANALYST	A.D.
BRIAN MINIX	LABORATORY ANALYST	WORKING ON B.S.
DIANE MOXLEY	LABORATORY ANALYST	B.S.
MELANIE NIESEN	LABORATORY AIDE	HIGH SCHOOL
SANDRA RILEY	LABORATORY ANALYST	B.S.

BARBARA RUBIO	LABORATORY ANALYST	M.S.
KIM WERNER	LABORATORY ANALYST	WORKING ON B.S.
MAL GROSS	ANALYTICAL SERVICES SALES & MARKETING	B.S.

II. LABORATORY FACILITIES

LAB SPACE

TOTAL SQUARE FEET = 7,500

BENCH SPACE

TOTAL LINEAR FEET = 200

SINKS

3- HOT AND COLD RUNNING WATER
5- COLD WATER SINKS FOR CONDENSING WATER

ELECTRICAL SERVICES

110 AND 220 VOLT SUPPLIES AVAILABLE

EXHAUST HOOD

FOUR HOODS EIGHT FEET IN LENGTH AVAILABLE.
100 FEET/MIN FACE VELOCITY EXHAUSTING.
COLD WATER, SINKS, AND GAS AVAILABLE IN HOODS.

HIGH PURITY WATER SUPPLY

CONTINENTAL WATER SYSTEM USING CARBON ABSORPTION, WATER SOFTENING, REVERSE OSMOSIS AND CATION/ANION EXCHANGE IN SERIES. FINAL WATER QUALITY IS 18 MEGOHM-CM. WATER QUALITY IS MONITORED DAILY AND INFORMATION IS USED TO DETERMINE THE NEEDS TO REGENERATE RESIN TANKS, WATER SOFTENER AND R/O SYSTEM.

COMPRESSED AIR

COMPRESSOR DELIVERS 1.8 CFM (50 L/MIN.) @ 60 PSI OF AIR FOR ATOMIC ABSORPTION INSTRUMENT AND IN-HOUSE AIR. COMPRESSOR IS MOUNTED ON 38 LITER TANK BUILT TO A.S.M.E. SPECIFICATIONS. AIR SUPPLY IS FILTERED TO REMOVE PARTICULATES AND OIL.

VACUUM

DIRECT DRIVE PUMP WITH AN EVACUATION RATE OF 50 LITERS/MIN IS USED FOR FILTRATION AND DESSICATION.

III. LABORATORY PRACTICES

GLASSWARE

All glassware used in the laboratory is borosilicate based KIMAX or PYREX. All volumetric flasks and pipets are Class A and meet NBS criteria.

SAMPLE BOTTLES

GLASS - Borosilicate glass with teflon lined screw caps

PLASTIC - Linear polyethylene with polypropylene screw caps

VOA VIALS - Glass with Teflon lined septa

GLASSWARE CLEANING

GENERAL - Glassware is cleaned in phosphate-free detergent, rinsed three times with tap water, and rinsed three times with deionized water.

PIPETS - Metal Pipets are soaked in a HNO_3 solution, wet chemical pipets are soaked in a soap solution, and rinsed 1 hour with deionized water in automatic pipet washer.

SPECIAL CLEANING

PHOSPHORUS - All glassware used in phosphorus test is washed with 1:1 hydrochloric acid and rinsed three times with deionized water.

METALS - All glassware used for metals testing is washed with 1:1 nitric acid and rinsed five times with deionized water.

Organics - All glassware is rinsed with acetone and high purity water.

CHEMICALS AND REAGENTS

All chemicals used in the laboratory are "Analytical Reagent Grade" unless another grade is specified in the methodology. Chemicals are dated upon receipt and discarded after shelf life is exceeded. Reagents made in the lab are stored in containers specified in methods. All purchased reagents, buffers, and standards are traceable to National Bureau of Standards.

IV. LABORATORY EQUIPMENT

ITEM	MAKE/MODEL	SPECIFICATIONS
ANALYTICAL BALANCES (2)	METTLER H 35 AR	160 gram capacity, (0.1 mg sensitivity)
TOP LOADING BALANCES (5)	METTLER PC 4400, PJ400	DUAL RANGE 4000 grams (0.1 gram sensitivity) 400 grams (0.01 gram sensitivity)
VISIBLE SPECTROPHOTOMETERS (2)	PYE/UNICAM -MODEL 330; -MODEL 8650	SINGLE BEAM, 350-900 MM, DIGITAL DISPLAY, ABSORBANCE OR CONCENTRATION
pH METERS (2)	-CORNING #130; -ORION RESEARCH	SENSITIVITY OF 0.001 PH UNIT, TEMPERATURE COMPENSATING
SPECIFIC ION METERS (2)	-CORNING #130 -ORION RESEARCH	SENSITIVITY OF 0.1 MV SENSITIVITY OF 0.01 MV
ATOMIC ABSORPTION SPECTROPHOTOMETERS (2)	PERKIN-ELMER #2380; #5000	-DOUBLE BEAM -MICROPROCESSOR CONTROL -BACKGROUND CORRECTOR
AA ATTACHMENTS	HGA-400 #023	-GRAPHITE FURNACE -RECORDER -ELECTRODELESS DISCHARGE SUPPLY
	MHS-10	-HYDRIDE SYSTEM MERCURY ANALYSIS SYSTEM
	#7300 WORKSTATION	MICROPROCESSOR COMPUTER
INDUCTIVELY COUPLED PLASMA SPECTROPHOTOMETER	PERKIN-ELMER #6500 WITH #7300 COMPUTER	SEQUENTIAL ANALYZER
AUTOANALYZER	LACHAT/QUIKCHEM	FLOW INJECTION ANALYSIS (FIA) SYSTEM
CONDUCTIVITY METER	YSI 33	
DRYING OVENS (3)	BLUE M#OV-510A-2	MECHANICAL CONVECTION, 50-260°C
	AMERICAN SCIENTIFIC	40-200°C
DESSICATORS (2)	LABCONCO 55300	-

HOT PLATES (2)	-LINDBERG 53014 -THERMOLYNE 2200	- -
MUFFLE FURNACE	BLUE M#M25A-2A	30-2000°F
STEAM BATHS (3)	PRECISION #66738	ELECTRICALLY HEATED
WATER BATHS (2)	BLUE M#MW1120A1	UP TO 100°C CONSTANT TEMPERATURE
VACUUM PUMP	PRECISION DD-50	DIRECT DRIVE, 50 LITERS/MIN
CENTRIFUGE	IEC SIZE 2, MODEL K	CAPACITY OF FOUR LITERS
REFRIGERATORS (7)	JORDAN FT-2-TR	47 CUBIC FOOT CAPACITY
SHAKER	EBERBACH 6000	UP TO 260 OSCILLATIONS/MIN.
THERMOMETER		NBS CERTIFIED
FLASH POINT TESTER	GCA/PRECISION SCIENTIFIC	
OXYGEN BOMB CALORIMETER	PARR BOMB #1341 PLAIN	
TOC INSTRUMENT	DOHRMANN DC-80 WITH AUTO SAMPLER	UV PROMOTED OXIDATION METHOD WITH IR DETECTOR FOR CO_2
GAS CHROMATOGRAPHS (4) (WITH AUTO SAMPLERS)	HEWLETT-PACKARD 5880 A, 5990,5890A TRACOR 540	ECD AND FID DETECTORS HALL AND PID DETECTORS
PURGE AND TRAPS (2)	TEKMAR 4000,4200	AUTO SAMPLER ATTACHMENTS
TOH INSTRUMENT	DOHRMANN ANALYZER	MICROCOULOMETRIC TITRATION

EQUIPMENT MAINTENANCE

All equipment is maintained according to manufacturer's recommendations. Ten major pieces of equipment are under the special maintenance programs detailed below.

ANALYTICAL BALANCES

The analytical balances are covered by a service contract. Once a year the instruments are completely cleaned and checked for accuracy by the manufacturer's service representative. In addition to the yearly service, a set of Class S-1 weights are available for periodic accuracy checks performed according to the guidelines given in ASTM, PART 41, 1976 entitled "Single Arm Balances Testing".

INDUCTIVELY COUPLED PLASMA (ICP), ATOMIC ABSORPTION (AA) AND GAS CHROMATOGRAPHS (GC)

The, ICP, AA, and GC instruments are covered by a manufacturer's service contract which calls for one to three visits per year for routine maintenance, cleaning, optics alignment, etc.

VISIBLE SPECTROPHOTOMETERS

The spectrophotometers are maintained according to manufacturer's guidelines. The wavelength alignment is checked monthly with colored reference solutions.

V. METHODOLOGY

ALL METHODS USED IN THE LABORATORY ARE USEPA APPROVED PROCEDURES OR STANDARD METHODS PROCEDURES DEPENDING ON THE REGULATORY REQUIREMENTS. REFERENCES ARE GIVEN BELOW:

1. "METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES" EPA-600/4-79-020 MARCH 1979 WITH TECHNICAL ADDITIONS EPA-600/4-82-055 DECEMBER 1982 AND EPA-600/4-84-017 MARCH 1984
2. USEPA TEST METHODS FOR ORGANIC POLLUTANTS. FEDERAL REGISTER OCTOBER 26, 1984. 40 CFR 136
3. "STANDARD METHODS FOR EXAMINATION OF WATER AND WASTEWATER" 16TH EDITION; APHA, AWWA, WPCF 1985.
4. "TEST METHODS FOR EVALUATING SOLID WASTES" SW-846, USEPA, 3RD EDITION, VOLUMES 1-A, B,C. AND D., NOVEMBER 1986

The following table summarizes each parameter run in the lab along with methods and references.

PARAMETER	METHOD USED	REFERENCE
ACID EXTRACTABLE ORGANICS	GAS CHROMATOGRAPHY	EPA #604, 40 CFR 136
ACIDITY	TITRIMETRIC	EPA 305.1
ALKALINITY	-TITRIMETRIC -COLORIMETRIC	EPA 310.1 EPA 310.2
ALUMINUM	-FLAME AA -ICP	EPA 200.1 EPA 200.7
AMMONIA NITROGEN	DISTILLATION, NESSLERIZATION	EPA 350.2
ANTIMONY	-FLAME AA -ICP	EPA 204.1 EPA 200.7
ARSENIC	GRAPHITE FURNACE AA	EPA 206.2
BARIUM	-FLAME AA -ICP	EPA 208.1 EPA 200.7
BERYLLIUM	-FLAME AA -ICP	EPA 210.1 EPA 200.7
BORON	CURCUMIN, COLORIMETRIC	EPA 213.3
CADMIUM*	-FLAME AA -ICP -GRAPHITE FURNACE AA	EPA 213.1 EPA 200.7 EPA 213.2
CALCIUM	-FLAME AA -ICP	EPA 215.1 EPA 200.7
CHLORIDE	COLORIMETRIC, FERRICYANIDE	EPA 325.2
CHROMIUM, HEXAVALENT	COLORIMETRIC	EPA 7196, SW-846
CHROMIUM, TOTAL*	-FLAME AA -ICP -GRAPHITE FURNACE AA	EPA 218.1 EPA 200.7 EPA 218.2
C.O.D.	COLORIMETRIC	EPA 410.4
COBALT	-FLAME AA -ICP	EPA 219.1 EPA 200.7
CONDUCTIVITY	WHEATSTONE BRIDGE	EPA 120.1

COPPER	-FLAME AA -ICP	EPA 220.1 EPA 200.7
CYANIDE, TOTAL	DISTILLATION, COLORIMETRIC	EPA 335.2
FLASH POINT	PENSKY-MARTENS, CLOSED CUP	EPA 1010, SW-846
FLUORIDE	SPECIFIC ION	EPA 340.2
HARDNESS, TOTAL	EDTA TITRIMETRIC -COLORIMETRIC	EPA 130.2 EPA 130.1
HERBICIDES	GAS CHROMATOGRAPHY	STANDARD METHODS 509B
IRON	-FLAME AA -ICP	EPA 236.1 EPA 200.7
LEAD*	-FLAME AA -ICP -GRAPHITE FURNACE	EPA 239.1 EPA 200.7 EPA 239.2
LITHIUM	FLAME AA	STANDARD METHODS 303A
MAGNESIUM	-FLAME AA -ICP	EPA 242.1 EPA 200.7
MANGANESE	-FLAME AA -ICP	EPA 243.1 EPA 200.7
MERCURY	COLD VAPOR FLAMELESS AA	EPA 245.1
MOLYBDENUM	FLAME AA	EPA 246.1
NICKEL	-FLAME AA -ICP	EPA 249.1 EPA 200.7
NITRATE NITROGEN	COLORIMETRIC, Cd REDUCTION	EPA 353.2
NITRITE NITROGEN	COLORIMETRIC	EPA 354.1
OIL & GREASE	GRAVIMETRIC,	EPA 413.1
pH	ELECTROMETRIC	EPA 150.1
PCB'S AND PESTICIDES	GAS CHROMATOGRAPHY	EPA #608 - 40 CFR 136
PHENOLS	DISTILLATION, COLORIMETRIC	EPA 420.2
PHOSPHORUS, ORTHO	ASCORBIC ACID COLORIMETRIC	EPA 365.2
PHOSPHORUS, TOTAL	PERSULFATE DIGESTION, ASCORBIC ACID COLORIMETRIC	EPA 365.2
POTASSIUM	FLAME AA	EPA 258.1

SELENIUM	GRAPHITE FURNACE AA	EPA 270
SILVER	-FLAME AA -ICP	EPA 272.1 EPA 200.7
SODIUM	FLAME AA	EPA 273.1
SOLIDS, DISSOLVED	GRAVIMETRIC, DRIED AT 180°C	EPA 160.1
SOLIDS, SUSPENDED	GRAVIMETRIC, DRIED AT 105°C	EPA 160.2
SOLIDS, TOTAL	GRAVIMETRIC, DRIED AT 105°C	EPA 160.3
SOLIDS, VOLATILE	GRAVIMETRIC, IGNITE AT 550°C	EPA 160.4
SULFATE	COLORIMETRIC, METHYLTHYMOL	EPA 375.2
SULFIDE	TITRIMETRIC	EPA 376.1
SURFACTANTS (MBAS)	COLORIMETRIC	EPA 425.1
THALLIUM	-FLAME AA -ICP	EPA 279.1 EPA 200.7
TIN	-FLAME AA -ICP	EPA 282.1 EPA 200.7
TOTAL KJELDAHL NITROGEN	DISTILLATION, NESSLERIZATION	EPA 351.3
TOTAL ORGANIC CARBON	UV OXIDATION	EPA 415.1
TOTAL ORGANIC HALOGEN	MICROCOULOMETRIC TITRATION	EPA 9020, SW-846
VANADIUM	FLAME AA	EPA 286.1
VOLATILE ORGANICS	PURGE AND TRAP, GAS CHROMATOGRAPHY	EPA #601, 602, 603- 40 CFR 136
ZINC	-FLAME AA -ICP	EPA 289.1 EPA 200.7

*GRAPHITE FURNACE METHOD USED FOR LOW LEVEL DRINKING WATER ANALYSIS

VI. SAMPLE COLLECTION, HANDLING AND PRESERVATION

PARAMETER	CONTAINER AND VOLUME REQUIRED	PRESERVATIVE	HOLDING TIME*
ACID EXTRACTABLE ORGANICS	AMBER GLASS, 1,000 ML	COOL, 4°C	7 DAYS, EXTRACTION 40 DAYS, AFTER EXTRACTION
ACIDITY	PLASTIC, 100 ML	COOL, 4°C	14 DAYS
ALKALINITY	PLASTIC, 100 ML	COOL, 4°C	14 DAYS
ALUMINUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
AMMONIA NITROGEN	PLASTIC, 500 ML	H_2SO_4 TO pH<2	28 DAYS
ANTIMONY	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
ARSENIC	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
BARIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
BERYLLIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
BORON	PLASTIC, 100 ML	COOL, 4°C	28 DAYS
CADMIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
CALCIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
CHLORIDE	PLASTIC, 100 ML	COOL, 4°C	28 DAYS
CHLORINATED ORGANICS	AMBER GLASS, 1,000 ML	COOL, 4°C	7 DAYS-EXTRACTION 40 DAYS-ANALYSIS
CHROMIUM, HEXAVALENT	PLASTIC, 200 ML	COOL, 4°C	24 HOURS
CHROMIUM, TOTAL	PLASTIC, 100 ML	HNO_3 to pH<2	6 MONTHS
C.O.D.	GLASS, 50 ML	H_2SO_4 TO pH<2	28 DAYS
COBALT	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
CONDUCTIVITY	PLASTIC, 100 ML	COOL, 4°C	28 DAYS
COPPER	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
CYANIDE, TOTAL	PLASTIC, 500 ML	NaOH TO pH>12	14 DAYS
FLASHPOINT	GLASS, 500 ML	COOL, 4°C	14 DAYS
FLUORIDE	PLASTIC, 100 ML	COOL, 4°C	28 DAYS

HARDNESS, TOTAL	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
HERBICIDES	AMBER GLASS, 1,000 ML	COOL, 4°C	7 DAYS, EXTRACTION 40 DAYS, AFTER EXTRACTION
IRON	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
LEAD	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
LITHIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
MANGNESIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
MANGANESE	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
MERCURY	GLASS, 200 ML	HNO_3 TO pH<2	28 DAYS
MOLYBDENUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
NICKEL	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
NITRATE NITROGEN	PLASTIC, 50 ML	Cool, 4°C	48 HRS
NITRITE NITROGEN	PLASTIC, 50 MLS	COOL, 4°C	48 HOURS
OIL & GREASE	GLASS, 500 ML	H_2SO_4 TO pH<2	28 DAYS
pH	PLASTIC, 50 ML	NONE	ANALYZE IMMEDIATELY
PCB'S AND PESTICIDES	AMBER GLASS, 1000 ML	COOL, 4°C	7 DAYS, EXTRACTION 40 DAYS, AFTER EXTRACTION
PHENOLS	GLASS, 500 ML	H_2SO_4 TO pH<2	28 DAYS
PHOSPHORUS, ORTHO	PLASTIC, 100 ML	COOL, 4°C	48 HOURS
PHOSPHORUS, TOTAL	PLASTIC, 100 ML	H_2SO_4 TO pH<2	28 DAYS
POTASSIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
SELENIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
SILVER	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
SODIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
SOLIDS, DISSOLVED	PLASTIC, 100 ML	COOL, 4°C	48 HOURS
SOLIDS, SUSPENDED	PLASTIC, 100 ML	COOL, 4°C	7 DAYS
SOLIDS, TOTAL	PLASTIC, 100 ML	COOL, 4°C	14 DAYS

SOLIDS, TOTAL	PLASTIC, 100 ML	COOL, 4°C	7 DAYS
SOLIDS, VOLATILE	PLASTIC, 100 ML	COOL, 4°C	7 DAYS
SULFATE	PLASTIC, 100 ML	COOL, 4°C	28 DAYS
SULFIDE	PLASTIC, 200 ML	ZINC ACETATE AND NaOH TO pH>9	7 DAYS
SURFACTANTS	GLASS, 500 ML	COOL, 4°C	48 HOURS
THALLIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
TIN	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
TOTAL KJELDAHL NITROGEN	PLASTIC, 500 ML	H_2SO_4 TO pH<2	28 DAYS
TOTAL ORGANIC CARBON	GLASS, 100 ML	H_2SO_4 TO pH<2	28 DAYS
TOTAL ORGANIC HALOGEN	AMBER GLASS, 1000 ML	COOL, 4°C	28 DAYS
VANADIUM	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS
VOLATILE ORGANICS	GLASS SEPTA VIAL 3 X 40 ML	COOL, 4°C	14 DAYS
ZINC	PLASTIC, 100 ML	HNO_3 TO pH<2	6 MONTHS

*In the October 26, 1984 (40 CFR 136), Federal Register, the EPA proposed these holding times for preserved samples.

Toxicity Leaching Tests

Methods used to perform EP toxicity tests correspond directly with EPA Method 1310 contained in SW-846. Quality control procedures which require duplicate and blank analysis every 20 samples are used. In addition, spiked and duplicate samples of the leachates are analyzed as required in RMT's quality control program.

The USEPA's new proposed Toxicity Characteristic Leaching Procedure or TCLP can be performed by the Laboratory. The procedures, equipment and analyses outlined in the June 13, 1986 Federal Register are followed. A quality control program of duplicates and blanks has been established to monitor the leaching procedure and USEPA methods are used in the analysis of all leachates.

VII. QUALITY CONTROL

The following quality control techniques are used to insure accurate results. All quality control data are documented and kept on file for inspection. More detailed information on each technique is found in the EPA Handbook for Analytical Quality Control in Water and Wastewater Labs, 1979.

REFERENCE SAMPLES

Quality control samples from the EPA are analyzed twelve times per year. The correct results are sent in sealed envelopes with the samples. The envelopes are opened only after the analysis is complete. The laboratory results are compared with the correct results and are available for inspection. The performance of the laboratory is reviewed after the analyses and any problem areas are defined and corrected.

STANDARD CURVES

With each new batch of reagents, a new standard curve is established using at least seven concentration levels and a blank. The new standard curve is compared to the existing curve and must agree within ±10%. The curve is kept on file for verification until a new curve is required. In each subsequent analysis run, the standard curve must be verified by a blank and two standards. The acceptance criteria for standard curve verification is ±10% for both standards.

The only exception to the above is atomic absorption work which uses three standards and a blank each time an analysis is run.

SPIKED ANALYSIS

Spikes are used to determine the accuracy of a given analysis. For each ten or less analyses performed, one sample is spiked and the percent recovery determined. The percent recovery data are compared to the quality control chart developed for each analysis (see below). The acceptance criteria for percent recovery is ± one standard deviation. The percent recovery is calculated as follows:

$$\text{STANDARDS)} \qquad \text{PERCENT RECOVERY} = 100 \; \frac{\text{OBSERVED VALUE}}{\text{KNOWN VALUE}}$$

$$\text{SPIKES)} \qquad \text{PERCENT RECOVERY} = 100 \; \frac{\text{OBSERVED VALUE-BACKGROUND VALUE}}{\text{SPIKE VALUE}}$$

DUPLICATE ANALYSIS

Duplicate analyses are used to determine the precision of each analysis. For each ten or less samples, one duplicate is run. The range between the duplicates is compared to the critical range value. The range must be below or equal to the critical range for acceptance. The critical range value is concentration dependent and determined by a large number of duplicate analyses.

STANDARD ADDITIONS

The method of standard additions is used primarily in furnace atomic absorption to determine interferences in different sample matrixes. Known amounts of a metal are added to the unknown sample at zero, one, two, and three times the expected amount. The results are plotted on a graph of absorbance vs. concentration and the value of the point of interception of the abscissa is the unknown concentration.

All furnace work requires the verification of matrix interferences before the need for standard additions can be determined. The unknown is spiked at a 1:4 dilution and compared to the unspiked result. If agreement is ±10%, then no interference exists, If it is greater than ±10%, then standard additions is required.

EP LEACH TEST

One out of every ten EP leach tests will have one duplicate and one blank analysis. The duplicate and blank data will be kept on file for inspection. It is difficult to establish acceptance criteria. A criteria of ±20% will be used until enough data is generated to determine higher or lower limits.

QUALITY CONTROL CHARTS

Charts are kept on each parameter and analyst. Accuracy and precision charts are available for inspection.

ACCURACY

When the results fall outside ±1 standard deviation or seven consecutive results are on the same side of the average percent recovery line, then the analysis is stopped and problem corrected. Then, the number of checks is doubled and all analyses in question are repeated or discarded.

PRECISION

When the precision of duplicate analyses is outside the critical range, the analysis is stopped and the problem corrected. The number of checks is doubled and all analyses in question are repeated or discarded.

Precision and accuracy data will be recorded daily on quality control charts. These charts will provide an easy evaluation of our work. We will use two types of charts for both precision and accuracy: cusum charts and Shewhart charts. One duplicate and spike should be run for each set of ten samples or less. The data will be recorded immediately after finishing the analyses, in both a table (see examples) and on the four quality control charts. Initially the data will have to be recorded in the table alone until there is sufficient data to prepare the charts, about 20 to 25 sets.

Cusum charts are based on a cumulative sum of the square of the difference between duplicates or the known and observed values of a spiked sample. The following equations are needed to prepare the charts for both precision and accuracy:

$$S_d^2 = \frac{\sum_{i=1}^{n} di^2 - \frac{\left(\sum_{i=1}^{n} di\right)^2}{N}}{N-1}$$

$$S_d = \sqrt{S_d^2}$$

$$S_o^2 = (0.8S_d)^2 = 0.64S_d^2$$

$$S_1^2 = (1.2S_d)^2 = 1.44S_d^2$$

$$UL(M) = \frac{2\log_e\left[\frac{1-\beta}{\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M\frac{\log_e\left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}}$$

$$LL(M) = \frac{2\log_e\left[\frac{\beta}{1-\alpha}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}} + M\frac{\log_e\left[\frac{S_1^2}{S_o^2}\right]}{\frac{1}{S_o^2} - \frac{1}{S_1^2}}$$

Where di = the difference between the ith set of duplicates or spiked samples

N = the total number of sets of duplicates or spiked samples used to construct the chart

S_d^2 = the variance of the differences

S_d = the standard deviation

S_o^2 = the minimum amount of variation allowed in the system

S_1^2 = the maximum amount of variation allowed in the system

α = the percent (in decimal fraction) of time we are willing to judge the procedure out of control when it is in control

β = the precent (in decimal fraction) of time we are willing to judge the precedure in control when it is out of control. The parameters α and β should be set between 0.05 and 0.15. A value of 0.05 gives a wide allowable range while a value of 0.15 gives a smaller range.

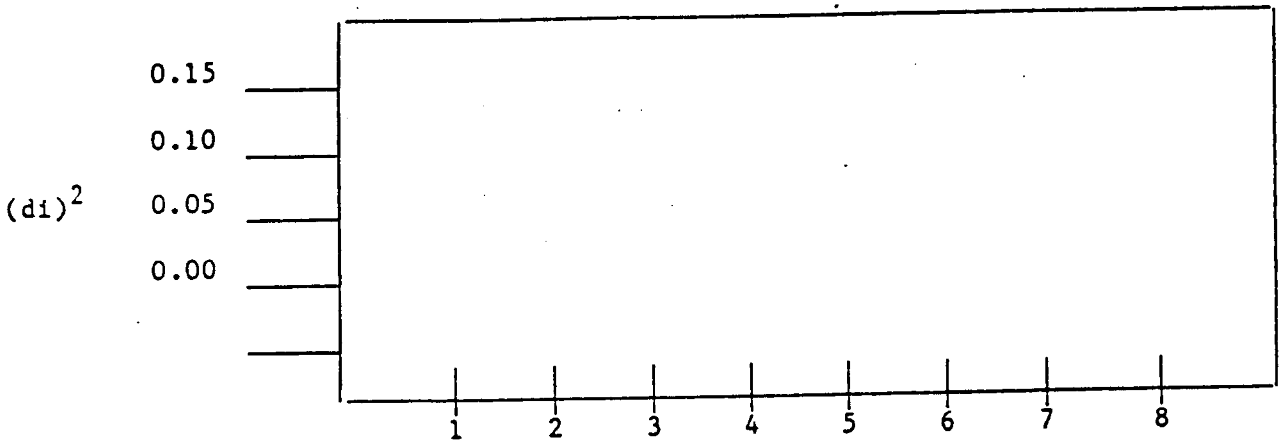
M = the number of sets of duplicates or spikes used in calculating the value to be plotted on the chart

UL (M) = the upper limit at M sets of samples

LL (M) = the lower limit at M sets of samples

The following is an example of the daily calculations used for precision data. For accuracy charts the same procedure is used except di is the difference between the known value of the spike and the observed value.

No. of duplicates (M)	Value 1	Value 2	di	di^2	$\Sigma(di)^2$
1	5.4	5.2	0.2	0.04	0.04
2	4.8	4.7	0.1	0.01	0.05
3	6.1	5.8	0.3	0.09	0.14



If a di^2 value falls out of control by the upper limit, the analysis will be stopped, the problem corrected, and the samples represented by the out of control value rerun sum. Data that falls out of control on the lower limit may be due to false reporting or an increase in precision. Analyses will be continued unless the trend changes. New control charts will be constructed based on recent data.

Shewhart charts use different calculations for accuracy and precision. The accuracy charts are based on percent recovery data. Percent recovery is calculated as

$$P = 100 \frac{\text{observed value}}{\text{known value}} \qquad \text{for standards}$$

and

$$P = 100 \frac{\text{observed} - \text{background}}{\text{spike}} \qquad \text{for spikes of real samples}$$

The standard deviation for percent recovery is then calculated:

$$\bar{P} = \frac{\sum_{i=1}^{N} P_i}{N}$$

$$S_p = \sqrt{\frac{\sum_{i=1}^{n} P_i^2 - \left(\sum_{i=1}^{n} P_i\right)^2 / N}{N-1}}$$

where N = the number of standards or spikes used

$\bar{P}$ = the average percent recovery

Sp = the standard deviation

Upper and lower control limits can be calculated from the following equations:

$UCL = \bar{P} + xS_p$ for the upper limit

$LCL = \bar{P} - xS_p$ for the lower limit

The value x determines how many standard deviations from the mean we are willing to call in control. To start our charts will have two sets of limits:

$UCL = \bar{P} + 1S_p$

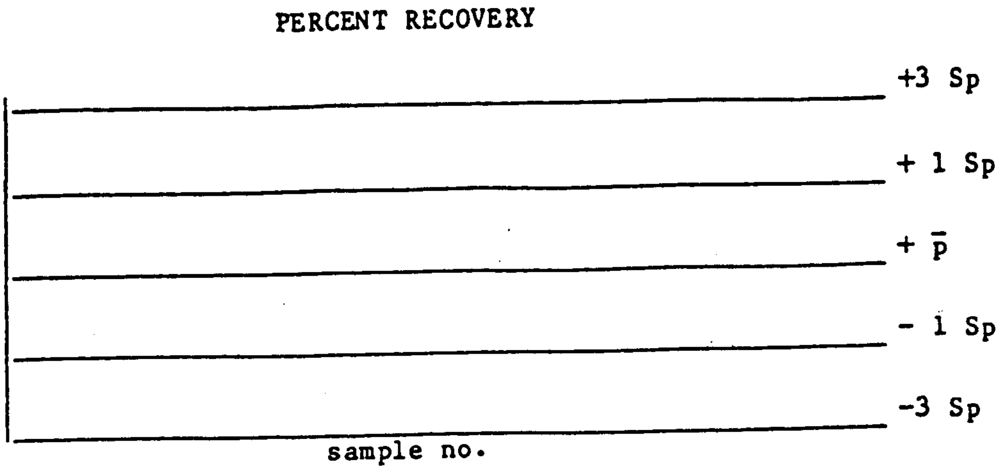
$UCL = \bar{P} - 1S_p$

and

$UCL = \bar{P} + 3S_p$

$LCL = \bar{P} - 3S_p$

On a daily basis the percent recovery of each spike is calculated and plotted on the chart.



The inside control limits (± 1Sp) will be used as a warning line. If data falls outside the outer control lines, the analyses will be stopped until the problem is corrected and the samples under question rerun.

The Shewhart chart for precision is based on the absolute value of the difference between each set of duplicate samples or their range. The average of a group of duplicates is calculated by

$$\bar{R} = \frac{\sum_{i=1}^{n} R_i}{N}$$

where N = the number of duplicates
R = the range of each set of duplicates (R is equal to di on the cusum charts)

The upper control limit can then be calculated

$$UCL_R = D_4\bar{R}$$

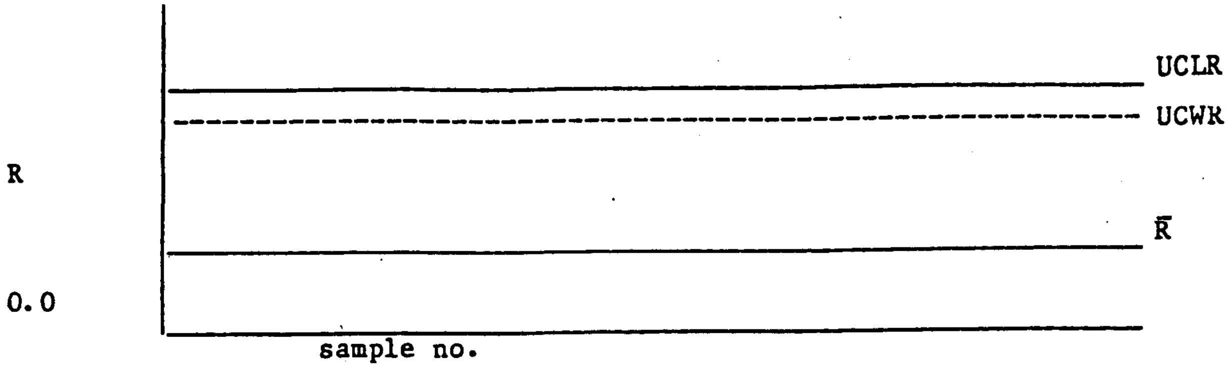
and the upper warning limit

$$UWL_R = 2/3\ (D_4\bar{R} - \bar{R}) + \bar{R}$$

where D4 is a constant dependent on the number of units in the subgroup. For duplicate samples there are two units, so D4 is equal to 3.27.

The upper warning limit (UWL_R) corresponds to the 95% confident level.

After the chart has been prepared, the range of each set of duplicates is calculated and plotted after every analysis.



The analysis will be stopped if the range of a set of duplicates exceeds the upper control limit

Precision For

Test: __________________________________

Method: __________________________________

Date	Set No. M	Value 1	Value 2	Range R or di	di^2	$\Sigma(di)^2$	Analyst	Comments

Accuracy For

Test: ______________________________

Method: ____________________________

Date	Set No. M	Conc. Added	Obs. Value	Backgrd Value	Obs.- Backgrd	% Recovery P	di	di^2	$\Sigma(di)^2$	Analyst	Comments

VIII. DATA HANDLING

All samples are recorded when they arrive at the laboratory. Samples are numbered and dated, and the required analyses are listed. Samples collected by RMT are labelled in the field. The labels contain the following information.

COLLECTION DATE:
COLLECTION TIME:
PLACE:
COLLECTOR:
SAMPLE DESCRIPTION:

A data sheet is written for each laboratory analysis. These data sheets contain the following information:

ANALYST:
ANALYTICAL METHOD:
RESULTS AND DATA:

All data and reports are kept on file for a minimum of three years. The data collected from quality control checks for each parameter are kept in a separate file.

CHAIN OF CUSTODY

All samples received in the lab must be accompanied by a sample record form (copy attached). This form is sent to the sampling site and is completed by sample collector. The chain of custody form is sealed inside the box containing the samples. The box is then shipped to the lab. All individuals who handle samples before shipment must sign off on a custody form. The sample shipper (UPS, etc.) does not sign the chain of custody. When the samples are received in the lab, the shipping container is inspected for signs of a broken seal. If a sample container has been opened before receipt by the lab, the sample will be refused and the chain of custody broken. If a sample container is still sealed, the chain of custody form will be completed by the lab and kept on permanent file.

Inc.
68 (D224)

RESIDUALS MANAGEMENT TECHNOLOGY, INC.
SAMPLE RECORD FORM

CLIENT: ____________________
JOB NUMBER: ____________________
LOCATION: ____________________
TYPE OF SAMPLES: ____________________
(WATER, WASTE WATER,
SOLID WASTE)

5. SAMPLING DATE: ____________________
6. SAMPLE COLLECTOR: ____________________
7. AFFILIATION: ____________________
8. SIGNATURE: ____________________
9. PURCHASE ORDER #: ____________________

COMMENTS ____________________

SAMPLE DESCRIPTION	RMT LABORATORY NUMBER	SAMPLE LOCATION AND DATE (R, LS, PS, D)*			

(REFRIGERATOR) LS (LABORATORY STORAGE) PS (PERMANENT STORAGE) D (DESTROYED)

SEQUENT ANALYSIS: ____ SAMPLE TEMP. ACCEPTABLE: ____ SAMPLE PRESERVED PROPERLY ____

PLES TRANSPORTED BY:

NAME: ____________________ DATE AND TIME: ____________________

AFFILIATION: ____________________ SIGNATURE: ____________________

MPLES RECEIVED BY:

NAME: ____________________ DATE AND TIME: ____________________

AFFILIATION: ____________________ SIGNATURE: ____________________

MPLE DESTRUCTION AUTHORIZATION:

SAMPLE NUMBERS: ____________________

PROJECT MANAGER: ____________________ DATE: ____________________

RMT PRINCIPAL: ____________________ DATE: ____________________

APPENDIX D

ALTERNATE GAS GENERATION PROCEDURE TO BE USED FOR FUTURE TESTING

Procedure For Acetylene Determination in Slag From Calcium Carbide Desulfurization

Equipment: Portable gas chromatograph

Suggested: Model OVA-128, Century Portable Organic Vapor Analyzer, Foxboro Analytical

Tedlar bag for connection to gas analyzer - about 30 liter capacity
250 or 500 ml beaker
Acetylene saturated water
Stirring Rod
Trip Scale
Acetylene gas source
Duct tape
Plastic or rubber glove

Preparation for Testing

1. Calibrate chromatograph with acetylene gas.
2. Prepare acetylene saturated water by bubbling acetylene through tap or DI water. Let water age for 2-4 hours after preparation before use.
3. Inflate tedlar bag to known volume with air-acetylene mixture. A wooden frame or box around bag will aid in achieving a known volume. Check instrument response and confirm calibration.
4. Cut slit in tedlar bag for inserting beaker, slag, etc. Slit is to be resealed after each insertion of materials.
5. Plastic glove should be permanently sealed into tedlar bag so sample manipulation can be performed without opening bag to atmosphere.

Testing Procedure

1. Unseal tedlar bag and put slag sample, (5-10 gms) beaker with acetylene saturated water in it and stirring rod inside.
2. Reseal bag with duct tape.
3. Insert hand into plastic glove and transfer slag sample into beaker containing acetylene saturated water.

4. Stir slag-water mixture vigorously for 2-3 minutes.
5. Read chromatograph after 15 to 30 minutes from mixing as time zero for ppm acetylene in bag.
6. Calculate acetylene volume produced after subtracting the reading for an equal volume of acetylene saturated water measured as a blank.

Example:

Reading for acetylene saturated water	80 ppm
Reading for slag after 15 minutes	360 ppm
Bag volume	30 liters
Slag weight	8.5 gms

$$(360 - 80)\ \text{ppm} \times \frac{30{,}000\ \text{ml}}{10^{6}\ \text{ml}} = 8.4\ \text{ml of acetylene}$$

8.4 ml ÷ 8.5 gms of sample = 0.99 ml/gm of slag

or

99 ml/100 gms of slag