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**CLOSURE PLAN FOR INTERIM STATUS
 HAZARDOUS WASTE CONTROL TANK**

SAGINAW NODULAR IRON CASTING PLANT
 GENERAL MOTORS CORPORATION
 SAGINAW, MICHIGAN

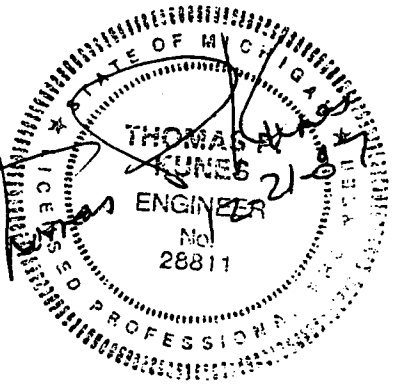
DECEMBER 1987

13

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 Casting 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. The RCRA 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 these four units is addressed in a separate Closure Plan.

This Closure Plan addresses the 1,1,1-Trichloroethane Waste Control Tank, located in the facility's Oil Building.

GMC-CFD Saginaw Nodular Iron identified and located the hazardous waste control tank as a 575-gallon rectangular tank located in the Oil Building on their November 17, 1980, Part A Permit Application. The hazardous waste control tank was actually used as an overflow and spillage collection tank during loading and unloading operations performed on 55-gallon drums of spent 1,1,1 trichloroethane degreasing solvent.

According to plant personnel, the hazardous waste control tank never accumulated enough liquid to require emptying during the entire period of its use.

This Closure Plan is based on achieving "clean closure" of the tank; therefore, no post-closure activities are expected. The Michigan Hazardous Waste Management Rules (Michigan Public Act 64) have adopted the 40 CFR Part 265 regulations by reference into the Michigan Administrative Code (R299.1103[1][m]). Where applicable, references will be made in this document to specific subsections of 40 CFR Part 265.

2. PURPOSE AND SCOPE

The purpose of this Closure Plan is to describe the closure activities that GMC-CFD Saginaw Nodular Iron will perform to close the 1,1,1-trichloroethane hazardous waste control tank.

The scope of this document is limited to providing a Closure Plan for the hazardous waste control tank; other solid waste management units are addressed in separate closure plans. This closure plan describes the following:

- Method of closure.
- Effect closure will have on the need for future maintenance and the potential for post-closure release of hazardous wastes and 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 Interim Status hazardous waste control tank at the GMC-CFD Saginaw Nodular Iron Plant, 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 J, and Michigan Act 64 have been addressed.

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

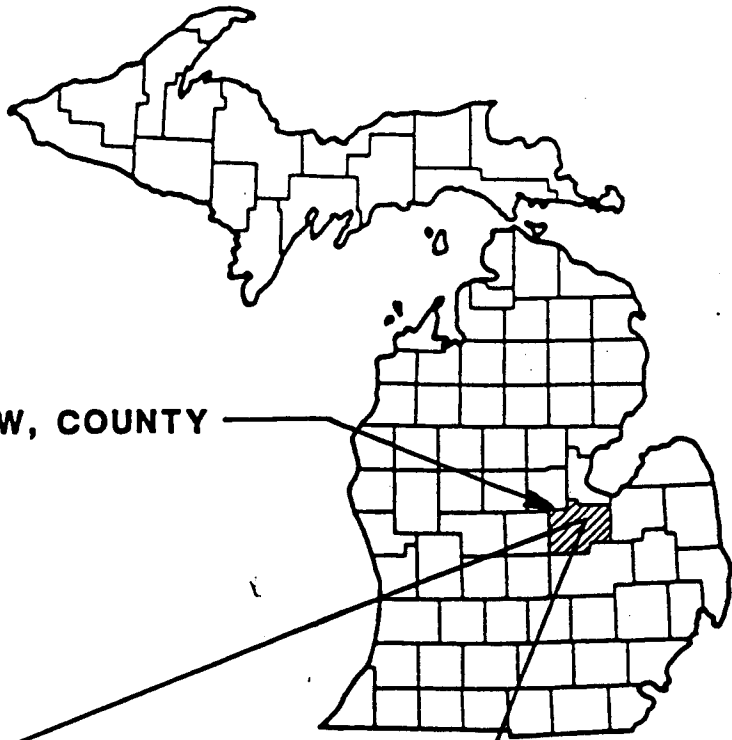
Contact: William Hudson, Environmental Coordinator

3.2 Site Description

Figure 1 shows the location of the GMC-CFD Saginaw Nodular Iron Plant on the Saginaw, Michigan USGS 7.5 minute map. The Oil Building is located south of the main foundry building. Figure 2 shows a plan view of the Oil Building and also shows the location of the hazardous waste control tank within the building.

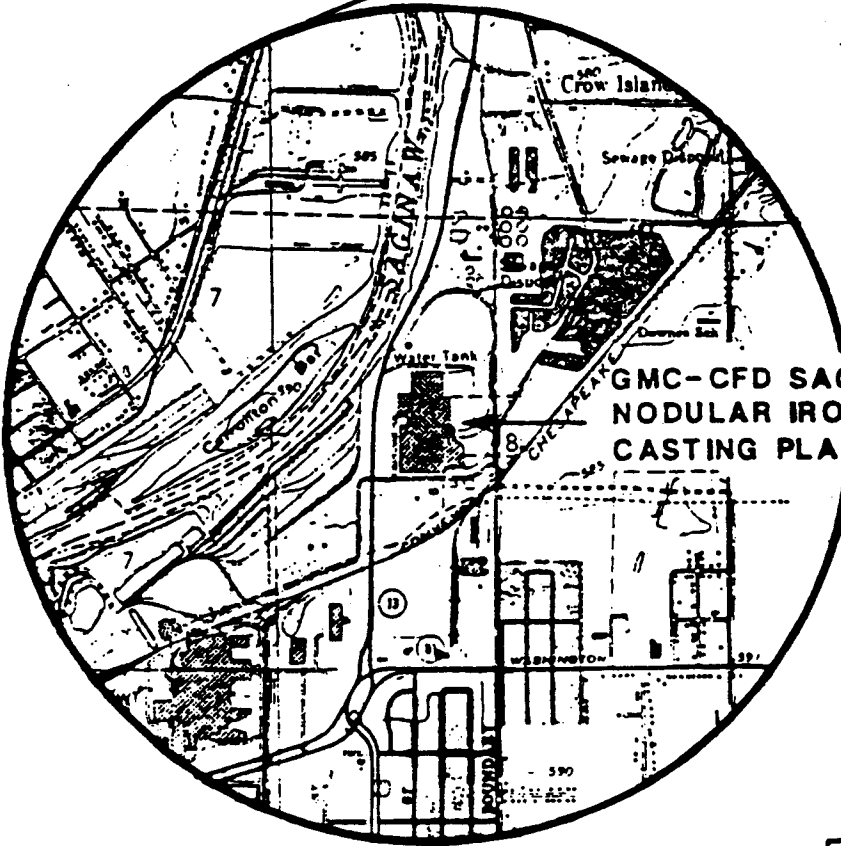
The hazardous waste control tank is a 575-gallon tank that is 71.5 inches long by 51.5 inches deep, with a liquid depth capacity of 36 inches. The tank is permanently fixed to the concrete floor slab of the Oil Building and has a metal strap grounding the tank within the building. Figure 3 is a photograph taken in August 1987 of the hazardous waste control tank.

The hazardous waste control tank was used to catch spills or leaks resulting from the loading and unloading of 55-gallon drums of spent 1,1,1 trichloroethane degreasing solvent. The 55-gallon drums were placed on the top of the tank (the tank has a screen top with a 3-inch-by-3-inch opening) and were filled or emptied only while on top of the tank. Leaks, spillage, or remaining spent solvent in pipes or hoses



SAGINAW, COUNTY

MICHIGAN

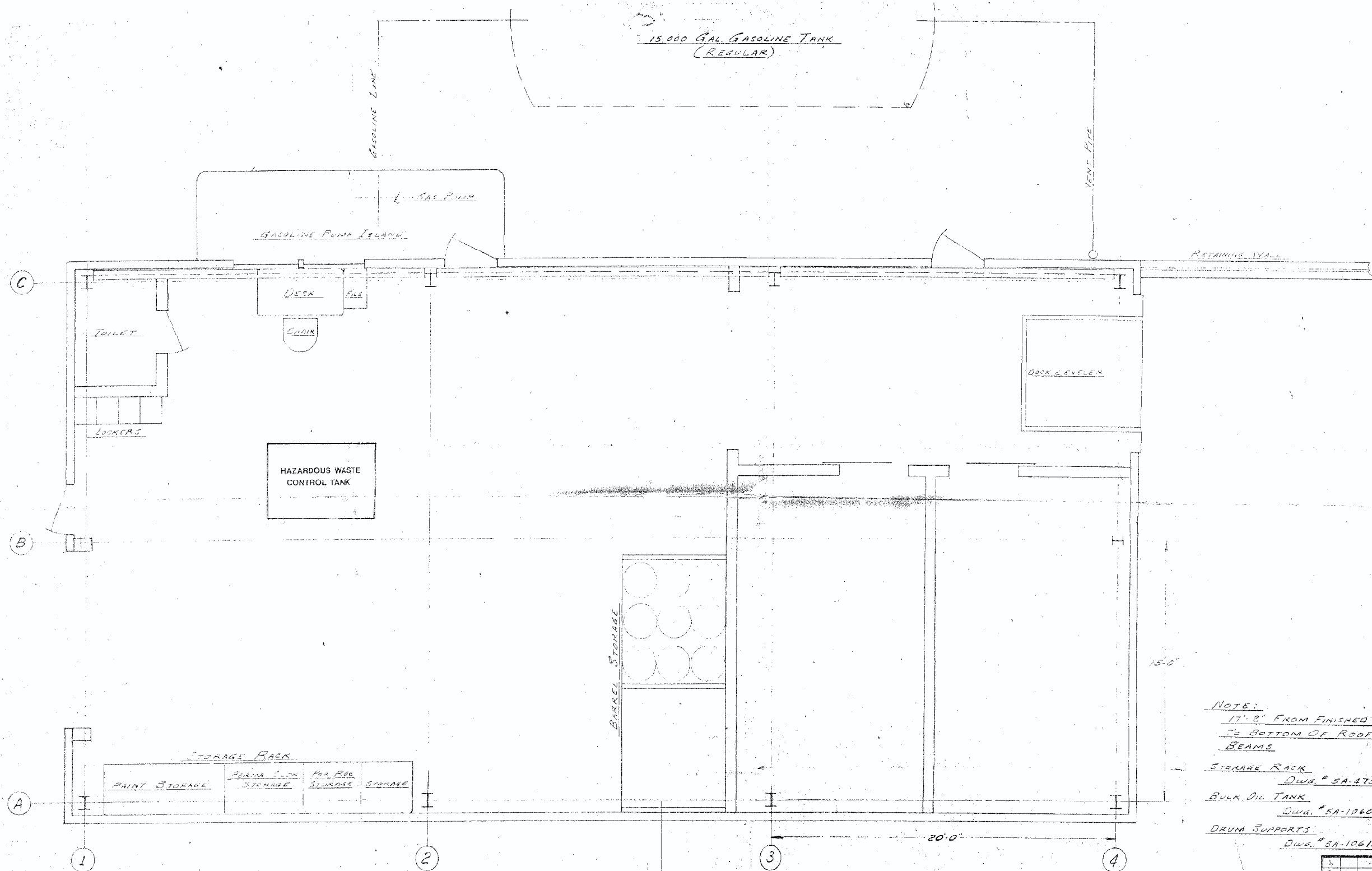


GMC-CFD SAGINAW
NODULAR IRON
CASTING PLANT

SAGINAW

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

FIGURE 1



NOTE:
 17'-8" FROM FINISHED FLOOR
 TO BOTTOM OF ROOF SUPPORT
 BEAMS

STORAGE RACK
 DWG. # 5A-475

BULK OIL TANK
 DWG. # 5A-10606

DRUM SUPPORTS
 DWG. # 5A-10618

NODULAR IRON FOUNDRY • SAGINAW FOUNDRIES
 PLANT NO. 1 SAGINAW, MICHIGAN DIVISION OF MOTOR DIVISION OF GENERAL MOTORS CORPORATION

DATE: 10-20-85
 SCALE: 1/4" = 1'-0"

APPROVED BY: [Signature]
 APPROVED BY: [Signature]

5A-440

3.				
2.				
1.				
NO.	BY	DATE	REVISION	APPR.
PROJECT: HAZARDOUS WASTE CONTROL TANK CLOSURE PLAN GM-CFD, SAGINAW, MICHIGAN				
SHEET TITLE: LOCATION OF TANK IN OIL BUILDING				
DRAWN BY: MCS		SCALE: 1/4" = 1'-0"		PROJ. NO: 1125.03
CHECKED BY: RCK		DATE: 10/24/87		DWG. NO:
APPROVED BY: TJU		DATE: 10/24/87		SHEET NO. OF: 2
DATE:				FIGURE 2
RMT				
Site 124 1005 East Westborough Ave. Warren, MI 48090 Phone: 696-2747				

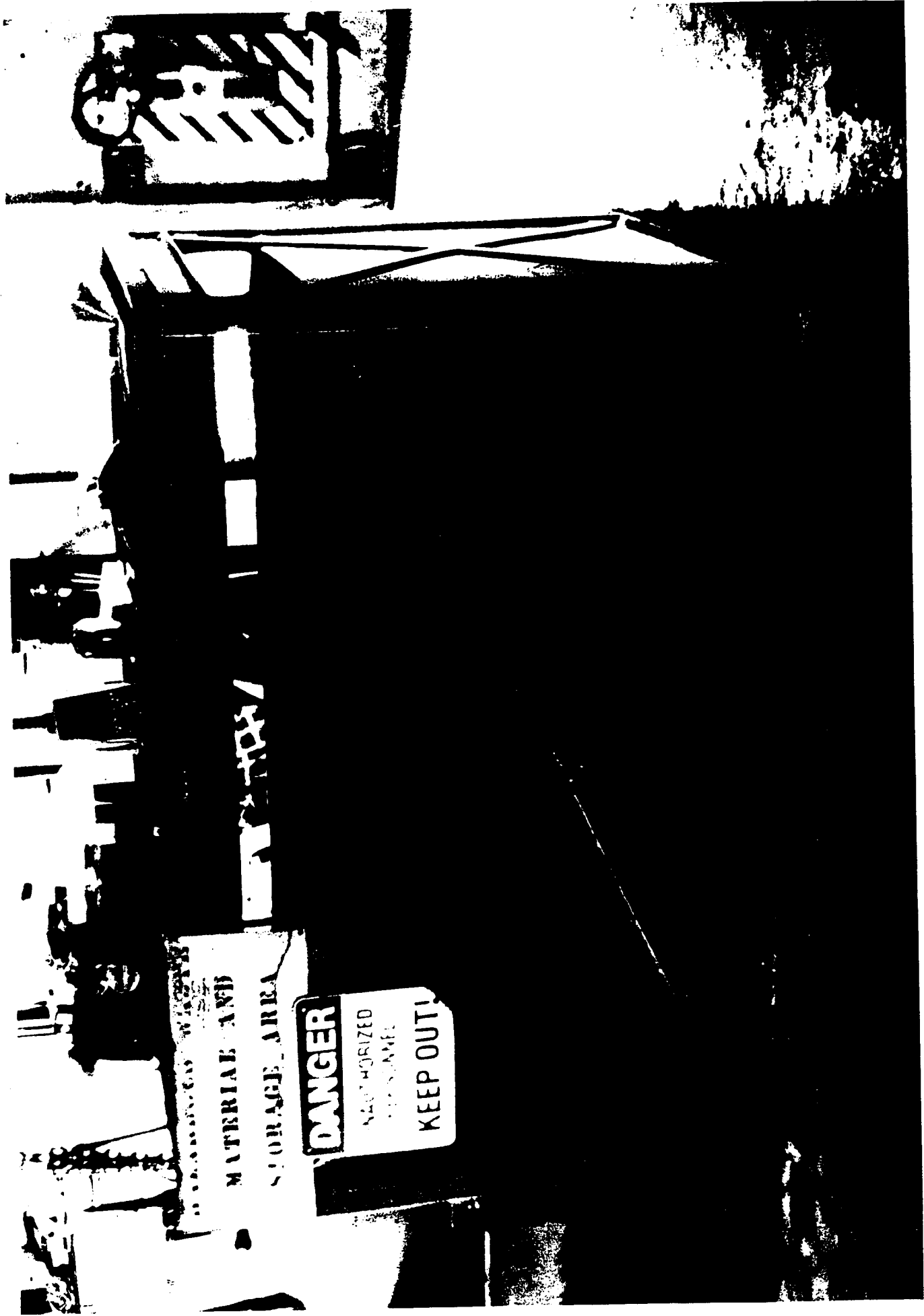


FIGURE 3 - Photograph of Hazardous Waste Storage Tank

were drained into the hazardous waste control tank. As stated previously, at no time during the operating history did the tank ever accumulate enough liquid to require emptying of the control tank.

Based on visual inspection by RMT personnel, both the tank and surrounding floor appear to be in good condition with no apparent evidence of cracks, leaks, or spills. No reportable spills or leaks occurred during the operation of the tank. In addition, none have been observed during any of the inspections performed by Michigan DNR personnel for the period 1983 to the present.

3.3 Waste Characterization

Spent 1,1,1 trichloroethane is a listed hazardous waste according to the definition of 40 CFR 261.31 with the USEPA hazardous waste number of F001. The product used by GMC-CFD Saginaw Nodular Iron is supplied under the trade name "Chlorothene VG" and is at least 94.5% by weight 1,1,1 trichloroethane.

Appendix A is the Material Safety Data Sheet for the 1,1,1 trichloroethane used as a degreasing solvent. Because no shipments of hazardous waste have been reported from the control tank, there are no hazardous waste manifests.

4. CLOSURE PERFORMANCE STANDARD

GMC-CFD Saginaw Nodular Iron must close the hazardous waste control tank in a manner that satisfies 40 CFR 265.111. Basically, the RCRA requirements indicate that closure 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:

- . the tank has been decontaminated; and
- . soils directly near the tank have not been impacted by 1,1,1 trichloroethane.

As already discussed in Section 3, observations by RMT personnel during August 1987 showed no evidence of spent degreasing solvent spills on the floor surrounding the hazardous waste control tank. In addition, no spills or leaks have been reported by plant personnel.

Based on the above information, GMC-CFD Saginaw Nodular Iron proposes that the following procedures serve as the closure performance standard for "clean" closure of the hazardous waste control tank:

1. The interior surface of the tank will be decontaminated with a mild alkaline detergent and rinsed with water from the City of Saginaw's potable water supply system. An alternative method may be used based on EPA 600/2-85-028 "Guide for Decontaminating Buildings, Structures, and Equipment at Superfund Sites." Wash and rinse water will be collected in barrels prior to discharge to the GMC-CFD Saginaw Nodular Iron wastewater treatment system. Three samples of the final rinse water will be collected from the barrels and analyzed for 1,1,1-trichloroethane. The wash and rinse activities will continue until the rinse water average concentration does not exceed 1.5 times the concentration measured in the trip blank or the raw water (whichever is greater) as described in Section 5.5.
2. The soil sampling and analysis plan, detailed in Section 5 of this closure plan, will then be implemented.
3. If the results of the soil sampling and analysis plan show that the hazardous waste control tank has not significantly affected the soils

below the tank (see Section 5.5), then closure will be considered complete, and documentation as described in Step 5 below will be provided.

4. If the soils below the spill tank area have been impacted, then the concrete will be removed and that soil material will be removed (from 1 to 2 1/2 feet or beyond) and disposed at an off-site hazardous waste disposal facility. Additional samples will then be collected until all underlying material indicates no environmental impact according to the criteria set in this Closure Plan.
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 GMC-CFD Saginaw Nodular Iron and an independent professional engineer, registered in the State of Michigan. The report will include chain-of-custody and laboratory analytical results.

5. SAMPLING AND ANALYSIS PLAN

5.1 Approach

Decontamination

The control tank was used to catch overflow and spillage of Chlorothene VG, which is 94.5% by weight 1,1,1 trichloroethane. Therefore, 1,1,1-trichloroethane is the best indicator parameter to determine contamination of the tank or surrounding soils. Because 1,1,1 trichloroethane is a volatile component, sampling and testing outside of the tank would not be indicative of contamination from the contents of the tank but most likely from the storage of unused 1,1,1 trichloroethane within the Oil Building.

The sampling and analysis plan described in this section describes decontamination of the control tank, and testing of the control tank decontamination rinse water for the presence of 1,1,1 trichloroethane. Wash and rinse liquids generated during decontamination will be collected in barrels. Prior to discharging these liquids to the GMC-CFD Saginaw Nodular Iron wastewater treatment system, three samples of the final decontamination rinsate will be collected and analyzed. A trip blank, field blank, and raw water sample from the City of Saginaw potable water supply will also be analyzed. Decontamination activities will continue until the average of the three final rinsate concentrations is less than 1.5 times the field blank or the raw water supply concentration (whichever is greater). At that point, decontamination of the control tank will be considered complete.

Soils

Visual observations of the concrete floor and records of the control tank operations indicate that controls and leaks have not occurred. One soil sample will be collected near the control tank by coring through the concrete floor. The location will be selected in a low-lying area or in an area of possible leakage from the tank. Four background soil samples will be collected from areas that have not been affected by the control tank or by specific use of 1,1,1-trichloroethane.

Compositional analysis (dry weight) will be performed (as described in USEPA Document SW-846) on the five soil samples. The concentration of 1,1,1-trichloroethane in the soil sample from the control tank area will be statistically compared (see Section 5.5) with the concentrations in the background samples. The comparison will be used to determine if operation of the control tank has affected the soil beneath the concrete floor.

5.2 Boring Locations

Both the control tank and the surrounding concrete floor appear to be in good condition, with no apparent evidence of cracks, leaks, or spills. There were no reportable leaks or spills during operation of the tank. Therefore, one soil sample will be collected beneath the floor by coring through the concrete floor. The boring will be located near the control tank, either in a low area or in the direction of surface liquid flow. The exact location will be determined based on site access, utilities, etc. The sample will be collected using a

split-spoon sampler.

In addition to the above boring, four background borings will be located as shown on Figure 4 (unless adequate data are available from previous background sample analyses). Samples from these four borings will be used to establish the range of background concentrations for 1,1,1-trichloroethane.

5.3 Sample Collection, Preservation, and Transport

Soils

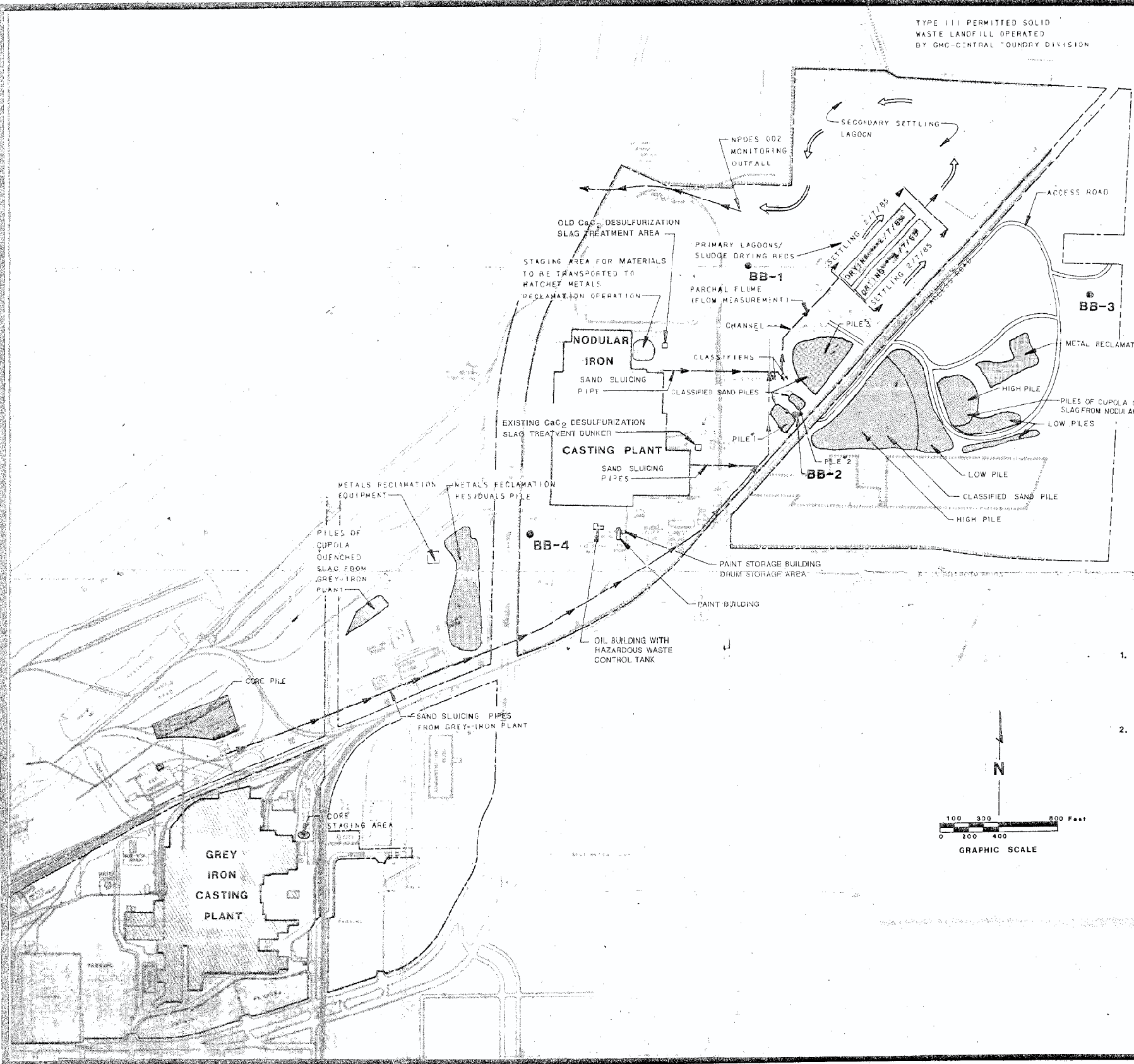
As previously described, one borehole will be drilled near the control tank using a coring device to penetrate the concrete, and four borings will be located in background areas. A visual observation will be made of the split-spoon samples and recorded on the boring log forms (Figure 5). Soil samples will be collected from the top 24 inches of soil using a split-spoon sampler as described in ASTM D1586-84.

Each of the five soil samples will be collected in duplicate in 40-milliliter VOA vials, by taking subsamples from the split-spoon sampler. Samples will be shipped with ice in coolers directly to the laboratory using chain-of-custody procedures. A chain-of-custody form is presented as Figure 6.

Liquids

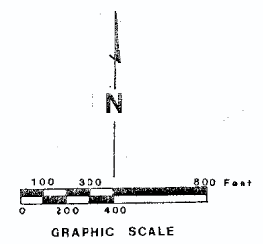
Wash and rinse waters used in decontaminating will be collected in barrels and disposed in the GMC-CFD wastewater treatment system (based on MDNR guidance). Prior to disposal, three samples will be collected of the final rinsate. These samples will be analyzed as described in

TYPE III PERMITTED SOLID
WASTE LANDFILL OPERATED
BY GMC-CENTRAL TOUNDRY DIVISION



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	APPROV.
PROJECT HAZARDOUS WASTE CONTROL TANK CLOSURE PLAN GM-CFD SAGINAW, MICHIGAN				
SHEET TITLE BACKGROUND SAMPLE LOCATIONS				
DRAWN BY EAP	SCALE	PROJ. NO. 1125.03	SHEET NO. OF	
CHECKED BY RCK	DATE PRINTED DEC 21 1987	DRAWING NO.	FIGURE 4	
APPROVED BY TJJ	DATE 10/15/87	RMT		

<p>LOG OF TEST BORING RMT, INC.</p> <p>PROJECT: _____ LOCATION: _____ DRILLED BY: _____ LOGGED BY: _____ DATE: _____</p>	<p>JOB NO.: _____ BORING NO.: _____ SURFACE ELEV.: _____ SHEET NO.: _____</p>
--	--

SAMPLE					VISUAL CLASSIFICATION and Remarks
Recovery		Moisture			
No.	Type	N	Depth	Depth	

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

Section 5.4. In addition, one field blank, one trip blank, and one raw water sample from the City of Saginaw potable water supply system will be analyzed.

Each of the six samples described above will be collected in triplicate in 40-milliliter VOA vials with teflon septum caps. The samples will be preserved with ice and transported to the laboratory using chain-of-custody procedures. A chain-of-custody form is presented as Figure 6.

Decontamination

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

- . All augers, coring machines, and other tools used in sampling will be steam-cleaned, or washed with soapy water and rinsed with potable water or by similar methods prior to use for each boring.
- . To avoid equipment contamination, all cleaning will be done at a site away from the sampling locations.
- . 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 cleaned 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.

5.4 Laboratory Analysis

Soils

The five soil samples will be analyzed for 1,1,1-trichloroethane as recommended in SW-846 (Test Methods for Evaluating Solid Wastes). Method 5030 (Purge and Trap) will be used for extraction, and Method 8010 (Halogenated Volatile Organic Compounds) will be used for analysis.

Liquids

The six liquid samples will be analyzed for 1,1,1-trichloroethane using Method 601 for purgeable hydrocarbons. Method 601 is a gas chromatographic procedure, and is described in 40 CFR Part 136 (Guidelines Establishing Test Procedures for the Analysis of Pollutants). Appendix B describes the QA/QC procedures required by the above-referenced analytical methods. If GMC-CFD Saginaw Nodular Iron elects to use another laboratory, equivalent QA/QC data will be submitted for MDNR approval.

5.5 Statistical Comparison

Soils

In order to determine if there has been an increase in 1,1,1 trichloroethane in the soils surrounding the waste control tank, 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 of 1,1,1-trichloroethane. This analysis will be performed for the data generated from the soil testing identified in Section 5.4. To determine if there has been an effect on surrounding soils, the analytical results of the soil sample from below the waste tank will be compared to the upper 99% confidence level value computed from the background soil sample results.

Liquids

For rinsate liquids, if the average of the three grab samples is less than 1.5 times the highest value of either the field blank or the raw water supply, decontamination will be considered complete. Otherwise, additional rinsing will be conducted. All wash and rinse liquids will be collected in barrels and disposed in the GMC-CFD Saginaw Nodular Iron wastewater treatment system, based on MDNR guidance.

6. HEALTH AND SAFETY

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 worker's 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 and Certification

The plan will be developed with the objective of complying with applicable federal, state, and local requirements.

The following references shall 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.

7. 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 any time during the life of the facility be reported. According to GMC-CFD Saginaw Nodular Iron personnel, the maximum amount of hazardous waste that was ever contained in the control tank was no greater than 50 gallons.

8. EQUIPMENT DECONTAMINATION

The equipment used in the tank decontamination activities may include rubber gloves, rubber boots, brushes, a pail, and a wet vacuum. The brushes, rubber gloves, and boots will be washed with soapy water and rinsed with potable water from the City of Saginaw water supply system. Water used for decontamination will be collected and disposed in the Saginaw Nodular Iron wastewater treatment system.

Soil sampling equipment will be decontaminated as described in Section 5.3. The liquid will be managed as described above for the decontamination equipment.

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

Once the GMC-CFD Saginaw Nodular Iron Plant ceases production and secures MDNR approval of this Closure Plan, GMC-CFD anticipates that the schedule outlined below will be implemented within 60 days and then completed within 180 days:

- . Contract for closure work.
- . Decontaminate control tank and obtain samples.
- . Analyze liquid and soil samples.
- . Prepare and submit Closure Documentation Report to the MDNR.

GMC-CFD estimates that closure will begin during 1988.

10. CLOSURE DOCUMENTATION

When closure is complete, the GMC-CFD Saginaw Nodular Iron Plant will submit a Closure Documentation Report to the Michigan DNR for approval. The closure activities will be observed by both plant personnel 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

MATERIAL SAFETY DATA SHEETS



CHEMCENTRAL/Detroit 13395 Huron River Drive Romulus, Michigan 48174 (313) 941-4800

February 12, 1987

Central Foundry Divison -GMC

Dear Customer:

Enclosed are Material Safety Data Sheet(s) for products you have purchased from us in the recent past, or are for products on which the Material Safety Data Sheet(s) have been revised since you last received them. Please consider them as the current copy to replace any previous version you may have received.

You are required effective May 25, 1986 by Federal Law CFR29, OSHA General Industry Standard 1910.1200 to make this information available to your employees.

We recognize that you may not be the person in your organization who most needs this information. If you are not, please direct it to your personnel who are responsible for the safe handling, storage, and disposal of products used in your facilities.

We value your business and want to be sure you have our current product safety information. If you need additional copies at any time, or have any questions concerning the information, please contact your CHEMCENTRAL/Detroit Corporation representative.

Thank you for your assistance,

Sincerely,

CHEMCENTRAL/Detroit Corporation

kah
encl.

MSDS enclosed for the following product(s):

1,1,1, TRICHLOROETHANE

BULK - 024424
DI - 024425 + 024429
DX - 024426
CAN - 024427 + 024428

MATERIAL SAFETY DATA SHEET

11/01/85

Dow Chemical U.S.A. Midland, MI 48674 Emergency Phone: 517-636-4400

MSD: 001111

Page: 1

PRODUCT NAME: CHLOROTHENE (R) SM SOLVENT

Effective Date: 10/04/85 Date Printed: 10/16/85 Product Code: 16896

1. INGREDIENTS:

1,1,1-Trichloroethane	CAS# 000071-55-6	95.5%
1,2-Butylene oxide	CAS# 000106-88-7	
Diethylene Ether	CAS# 000123-91-1	
Nitromethane	CAS# 000075-52-5	

The hazard information presented is based on tests conducted on this or similar mixtures. Therefore, pursuant to the OSHA Hazard Communication Standard (see 29 CFR Part 1910.1200 (g) (2) (B)), the information is based on the tested mixture and not individual ingredients.

2. PHYSICAL DATA:

BOILING POINT: 165F (74C)
VAP PRESS: 100 mmHg @ 20C
VAP DENSITY: 4.55
SOL. IN WATER: 0.07 g/100g @ 25C
SP. GRAVITY: 1.321 @ 25/25C
APPEARANCE: Colorless liquid.
ODOR: Irritating odor at high concentrations.

3. FIRE AND EXPLOSION HAZARD DATA:

FLASH POINT: None
METHOD USED: TOC, TCC, CDC

FLAMMABLE LIMITS
LFL: 7.5% @ 25C
UFL: 15% @ 25C

EXTINGUISHING MEDIA: Water fog.

FIRE & EXPLOSION HAZARDS: Not available.

(Continued on Page 2)

(R) Indicates a trademark of The Dow Chemical Company

MATERIAL SAFETY DATA SHEET

Dow Chemical U.S.A. Midland, MI 48674 Emergency Phone: 517-636-4400

MSD: 001111 Page: 2

PRODUCT NAME: CHLOROTHENE (R) SM SOLVENT

Effective Date: 10/04/85 Date Printed: 10/16/85 Product Code: 16896

3. FIRE AND EXPLOSION HAZARD DATA: (CONTINUED)

FIRE-FIGHTING EQUIPMENT: Self-contained, positive pressure respiratory equipment.

4. REACTIVITY DATA:

STABILITY: (CONDITIONS TO AVOID) Avoid open flames, welding arcs or other high temperature sources which induce thermal decomposition.

INCOMPATIBILITY: (SPECIFIC MATERIALS TO AVOID) Water - long term contact can deplete stabilizers followed by slow hydrolysis producing corrosive acid. Avoid prolonged contact with, or storage in, aluminum or its alloys. Metallic aluminum and zinc powders should be avoided.

HAZARDOUS DECOMPOSITION PRODUCTS: Hydrogen chloride and very small amounts of phosgene and chlorine.

HAZARDOUS POLYMERIZATION: Will not occur.

5. ENVIRONMENTAL AND DISPOSAL INFORMATION:

ACTION TO TAKE FOR SPILLS/LEAKS: Small leaks: Mop up, wipe up, or soak up immediately. Remove to out-of-doors.
Large spills: Evacuate area. Contain liquid; transfer to closed metal containers. Keep out of water supplies.

DISPOSAL METHOD: When disposing of the unused contents, the preferred options are to send to licensed reclaimer, permitted incinerators, or to evaporate small quantities in compliance with local, state, and federal regulations including Subtitle C of the Resource Conservation and Recovery Act. Dumping into sewers, on the ground, or into any body of water is strongly discouraged, and may be illegal. Consult The Dow Chemical Company for further information.

(Continued on Page 3)

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MATERIAL SAFETY DATA SHEET

Dow Chemical U.S.A. Midland, MI 48674 Emergency Phone: 517-636-4400

MSD: 001111

Page: 3

PRODUCT NAME: CHLOROTHENE (R) SA SOLVENT

Effective Date: 10/04/85 Date Printed: 10/16/85 Product Code: 16896

6. HEALTH HAZARD DATA:

EYE: May cause pain. May cause slight transient (temporary) irritation with slight transient corneal injury. Vapors may irritate eyes.

SKIN CONTACT: Prolonged or repeated exposure may cause skin irritation. Repeated contact may cause drying or flaking of skin.

SKIN ABSORPTION: A single prolonged skin exposure is not likely to result in absorption of harmful amounts. The LD50 for rabbits is about 15,000 mg/kg.

INGESTION: Single dose oral toxicity is low. The LD50 for rats is >10,000 mg/kg. If aspirated (liquid enters the lung), may be rapidly absorbed through the lungs and result in injury to other body systems.

INHALATION: Minimal anesthetic or narcotic effects may be seen in the range of 500-1000 ppm trichloroethane. Progressively higher levels over 1000 ppm may cause dizziness, drunkenness; concentrations as low as 10,000 ppm can cause unconsciousness and death. In confined or poorly ventilated areas, vapors which readily accumulate can cause unconsciousness and death. These high levels may also cause cardiac arrhythmias (irregular heartbeats).

SYSTEMIC & OTHER EFFECTS: Based on available data, repeated exposures are not anticipated to cause any significant adverse effects. Similar formulations did not cause cancer in long-term animal studies. Birth defects are unlikely. Exposures having no adverse effects on the mother should have no effect on the fetus. In animal studies, has been shown not to interfere with reproduction. Results of in vitro ("test tube") mutagenicity tests have been inconclusive. Results of mutagenicity tests in animals have been negative.

(Continued on Page 4)

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MATERIAL SAFETY DATA SHEET

Dow Chemical U.S.A. Midland, MI 48674 Emergency Phone: 517-636-4400

MSD: 001111

Page: 4

PRODUCT NAME: CHLOROTHENE (R) SM SOLVENT

Effective Date: 10/04/85 Date Printed: 10/16/85 Product Code: 16896

7. FIRST AID:

EYES: Irrigate immediately with water for at least 5 minutes.

SKIN: Wash off in flowing water or shower. Remove contaminated clothing and wash before reuse.

INGESTION: Do not induce vomiting. Call a physician and/or transport to emergency facility immediately.

INHALATION: Remove to fresh air. If not breathing, give mouth-to-mouth resuscitation. If breathing is difficult, give oxygen. Call a physician.

NOTE TO PHYSICIAN: Because rapid absorption may occur through lungs if aspirated and cause systemic effects, the decision of whether to induce vomiting or not should be made by an attending physician. If lavage is performed, suggest endotracheal and/or esophageal control. Danger from lung aspiration must be weighed against toxicity when considering emptying the stomach. Exposure may increase "myocardial irritability." Do not administer sympathomimetic drugs unless absolutely necessary. No specific antidote. Supportive care. Treatment based on judgment of the physician in response to reactions of the patient.

8. HANDLING PRECAUTIONS:

EXPOSURE GUIDELINE(S): 1,1,1-Trichloroethane - OSHA standard is 350 ppm and current ACGIH TLV is 350 ppm (450 ppm STEL).

ACGIH TLV is 25 ppm skin for diethylene ether; the STEL is 100 ppm. OSHA PEL is 100 ppm skin for diethylene ether. Dow Industrial Hygiene Guide for 1,2-butylene oxide is 40 ppm (excursion 100 ppm). ACGIH TLV for nitromethane is 100 ppm with a STEL of 150 ppm.

VENTILATION: Control airborne concentrations below the exposure guideline. Use only with adequate ventilation. Local exhaust ventilation may be necessary for some operations. Lethal concentrations may exist in areas with poor ventilation.

(Continued on Page 5)

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MATERIAL SAFETY DATA SHEET

Dow Chemical U.S.A. Midland, MI 48674 Emergency Phone: 517-636-4400

MSD: 001111 Page: 5

PRODUCT NAME: CHLOROTHENE (R) SM SOLVENT

Effective Date: 10/04/85 Date Printed: 10/16/85 Product Code: 16896

8. HANDLING PRECAUTIONS: (CONTINUED)

RESPIRATORY PROTECTION: Atmospheric levels should be maintained below the exposure guideline. When respiratory protection is required for certain operations, use an approved air-purifying respirator. For emergency and other conditions where the exposure guideline may be greatly exceeded, use an approved positive pressure self-contained breathing apparatus. In confined or poorly ventilated areas, use an approved positive pressure self-contained breathing apparatus.

SKIN PROTECTION: For brief contact, no precautions other than clean body-covering clothing should be needed. When prolonged or frequently repeated contact could occur, use protective clothing impervious to this material. Selection of specific items such as gloves, boots, apron, or full body suit will depend on operation.

EYE PROTECTION: Use safety glasses. Where contact with liquid is likely, chemical goggles are recommended because eye contact with this material may cause pain, even though it is unlikely to cause injury.

9. ADDITIONAL INFORMATION:

SPECIAL PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE: Handle with reasonable care. Avoid breathing vapors. Store in a cool dry place. Concentrated vapors of this product are heavier than air and will collect in low areas such as pits, degreasers, storage tanks, and other confined areas. Do not enter areas where vapors of this product are suspected unless special breathing apparatus is used and an observer is present for assistance.

1,1,1-Trichloroethane products should not be packaged in aluminum aerosol cans or with finely divided aluminum or its alloys in an aerosol can.

Aluminum is not an acceptable material of construction for pumps, mixers, fittings, storage tanks for 1,1,1-trichloroethane

(Continued on Page 6)

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MATERIAL SAFETY DATA SHEET

Dow Chemical U.S.A. Midland, MI 48674 Emergency Phone: 517-636-4400

MSD: 001111

Page: 6

PRODUCT NAME: CHLOROTHERE (R) SM SOLVENT

Effective Date: 10/04/85 Date Printed: 10/16/85 Product Code: 16896

9. ADDITIONAL INFORMATION: (CONTINUED)

products or formulations. Metallic aluminum and zinc powders should be avoided. For additional information on toxicity, handling precautions, and first aid, refer to chlorinated solvents literature form no. 100-5792.

MSDS STATUS: Revised sections 1, 5, 6, 8, and 9.

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The information herein is given in good faith, but no warranty, expressed or implied, is made. Consult The Dow Chemical Company for further information.

APPENDIX B

RMT 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

<u>STAFF MEMBER</u>	<u>TITLE</u>	<u>ACADEMIC</u>
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

<u>ITEM</u>	<u>MAKE/MODEL</u>	<u>SPECIFICATIONS</u>
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 MHS-10 #7300 WORKSTATION	-GRAPHITE FURNACE -RECORDER -ELECTRODELESS DISCHARGE SUPPLY -HYDRIDE SYSTEM MERCURY ANALYSIS SYSTEM 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 AMERICAN SCIENTIFIC	MECHANICAL CONVECTION, 50-260 °C 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.

<u>PARAMETER</u>	<u>METHOD USED</u>	<u>REFERENCE</u>
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

<u>PARAMETER</u>	<u>CONTAINER AND VOLUME REQUIRED</u>	<u>PRESERVATIVE</u>	<u>HOLDING TIME*</u>
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 ₂ SO 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 ₂ SO 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 ML	COOL, 4°C	48 HOURS
OIL & GREASE	GLASS, 500 ML	H ₂ SO 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 ₂ SO ₄ TO pH<2	28 DAYS
PHOSPHORUS, ORTHO	PLASTIC, 100 ML	COOL, 4°C	48 HOURS
PHOSPHORUS, TOTAL	PLASTIC, 100 ML	H ₂ SO 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 ₂ SO ₄ TO pH<2	28 DAYS
TOTAL ORGANIC CARBON	GLASS, 100 ML	H ₂ SO 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 $\pm 10\%$. 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 $\pm 10\%$ 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 \pm one standard deviation. The percent recovery is calculated as follows:

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

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

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 $\pm 10\%$, then no interference exists, If it is greater than $\pm 10\%$, 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 $\pm 20\%$ 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:

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

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

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

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

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

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

Where d_i = the difference between the i th 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

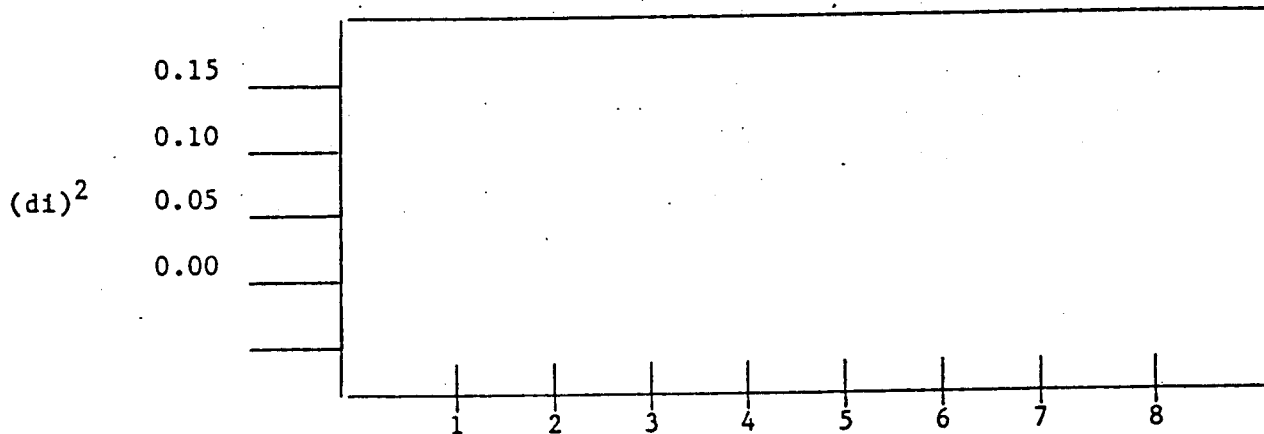
- α = the percent (in decimal fraction) of time we are willing to judge the procedure out of control when it is in control
- β = the percent (in decimal fraction) of time we are willing to judge the procedure in control when it is out of control. The parameters α and β should be set between 0.05 and 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 d_i is the difference between the known value of the spike and the observed value.

<u>No. of duplicates (M)</u>	<u>Value 1</u>	<u>Value 2</u>	<u>d_i</u>	<u>d_i^2</u>	<u>$\Sigma(d_i)^2$</u>
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 d_i^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

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

and

$$P = 100 \frac{\text{observed} - \text{background}}{\text{spike}}$$

for standards

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

S_p = the standard deviation

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

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

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

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:

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

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

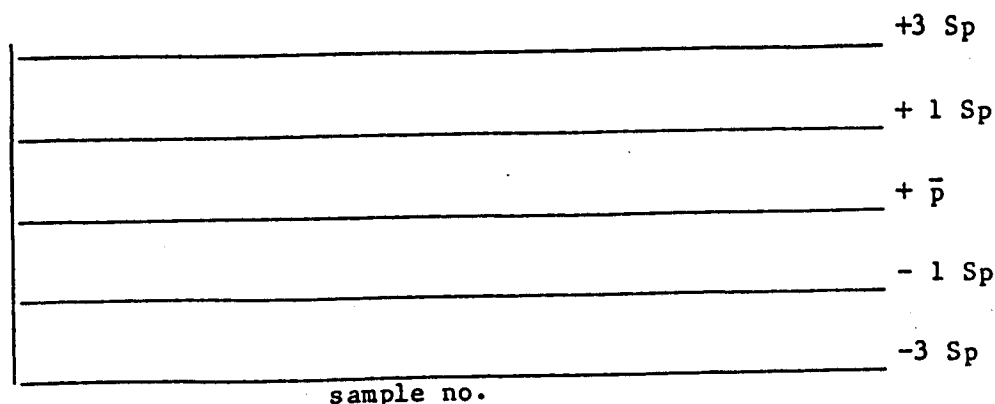
and

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

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

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

PERCENT RECOVERY



The inside control limits ($\pm 1Sp$) 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

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

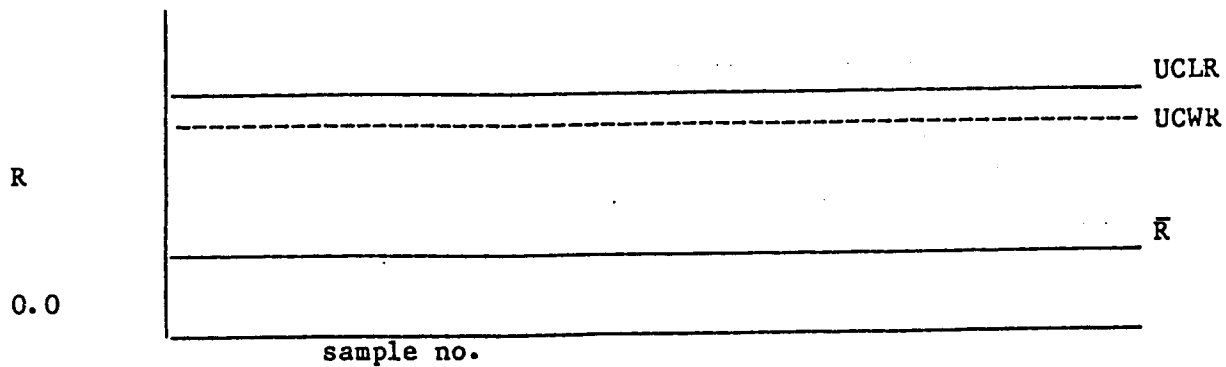
and the upper warning limit

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

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

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.

