US EPA RECORDS CENTER REGION 5
1004851

MICHIGAN DEPARTMENT OF NATURAL RESOURCES ACT 64 OPERATING LICENSE APPLICATION

Detrex Corporation
Detroit, Michigan
August 11, 1988 (Original Submission)
June 24, 1991 (Re-Submission)

RECEIVED
JUN 26 1991
Waste Management Division

JUNE 1991
REF. NO. 2471 (1)

CONESTOGA-ROVERS & ASSOCIATES

MICHIGAN DEPARTMENT OF NATURAL RESOURCES

INTEROFFICE COMMUNICATION

July 17, 1991

TO: Ronda Hall, Permits, WMD

FROM: Nadine Romero, Geotech Unit, WMD

Nadine Romero

SUBJECT: Detrex Corporation Act 64 Permit Application
Revisions of June 24, 1991
Gold Shield Solvents, Detroit
MID 091 605 972

I reviewed the Detroit Detrex Act 64 Permit Application as revised on June 24, 1991. The following concerns and deficiencies are noted and should be addressed in your meeting with the facility scheduled for July 18, 1991:

1. In the revised application, the Detrex facility requests waivers for environmental/ground water monitoring on the basis that facility storage activities take place inside a structure with secondary containment and that a peripheral curb around the concrete unloading area and joint/crack resealing will be done. However, Section D of the application as cited in the Waiver request on page E-9 does not give specific details on the construction of the secondary containment for the truck unloading area. If adequate secondary containment can be built for the truck unloading area, then a waiver from ground water monitoring may be justified.

2. As outlined in my February 2, 1991 memo, it is not clear where the grain size analyses are for the CL designations on the boring logs. This item still needs to be addressed.

3. Contingent on the permit issuance and a ground water monitoring waiver, Detrex should verify the quality of ground water in the ground water monitoring well located east of the building, providing a one time set of analytical results for the file.

I have no other comments at this time. If Detrex can provide adequate secondary containment, then I see no reason why they cannot get a ground water monitoring waiver. Please see me if you have any concerns or comments.

cc: De Montgomery/Geotech Unit
HW Permits C&E File

MICHIGAN DEPARTMENT OF NATURAL RESOURCES

INTEROFFICE COMMUNICATION

February 2, 1991

TO: Ronda Hall, Permits, WMD

FROM: Nadine Romero, Geotech Unit, WMD

SUBJECT: Detrex Corporation Act 64 Permit Application
Gold Shield Solvents, Detroit
MID 091 605 972

I have completed a technical review of the Detrex Gold Shield Solvents, Detroit facility Act 64 permit application. The Detrex facility conducted a hydrogeologic study in April, 1989 completing 3 soil borings for the 1 acre site. This facility requests an environmental monitoring waiver under P.A. 64, 1979 as ammended, R299.9611(3), based on the hydrogeologic findings and their contention that waste handling activities take place inside or under a structure.

Because unloading of hazardous wastes drums occurs in an unprotected or open loading dock that is not secondarily contained, the facility will have to provide a proposal for some type of environmental monitoring. The difficulty with this site is that 3 to 4 feet of sand and gravel fill overlies aproximately 90 to 100 feet of silty clay, a common lithology for this area of Detroit. Ground water was encountered in a sand unit at 100 feet below ground surface. Thus, a ground water monitoring program capable of detecting hazardous waste constituents from the storage facility may not be plausible. Regardless, the facility is going to have to come up with some type of proposal to provide detection monitoring of the soils and ground water of this site [in accordance with R299.9611 and R299.9612] or upgrade the unloading area with curbs, paving and a sump (secondary containment).

In summary the following deficiencies and concerns are cited:

1. It is not clear from the hydrogeologic report why additional

split spoon samples were not analyzed for grain size distribution and classified under the unified soil classification. Boring logs are missing information from the grain size analysis results i.e. boring BH2-89 denotes "sandy" giving inference to some sand in the clay from 33-36 feet, however, grain size analysis shows this unit is a silty sand (SM) and not a silty clay. Boring log BH3-89 also does not appropriately record a silty sand (SM) at the 33-36 foot interval. In fact, there are no grain size analyses for the clay (CL) classification as cited on the boring logs. [R299.9506(2)(a)(ii)].

2. The hydrogeological report lacks a topographic map delineating the waste management area and the boring and well locations [R299.9506(1)(e)].

3. The hydrogeological report will need to propose a ground water or environmental monitoring program capable of detecting any releases of hazardous waste or hazardous waste constituents from the facility [R299.9506(2); R299.9611(1) and (2)].

This concludes my review of the Act 64 permit application for the Detroit Detrex facility. If you have any questions please see me, I would like to discuss components of this review as well.

cc: Liz Browne/Geotech Unit
HW Permits C&E File

NATURAL RESOURCES COMMISSION
THOMAS J. ANDERSON
MARLENE J. FLUHARTY
GORDON E. GUYER
KERRY KAMMER
O. STEWART MYERS
DAVID D. OLSON
RAYMOND POUPORE

STATE OF MICHIGAN

JAMES J. BLANCHARD, Governor

DEPARTMENT OF NATURAL RESOURCES
Gordon E. Guyer, Director

S.E. MICHIGAN FIELD OFFICE
Waste Management Division
505 W. Main
Northville, MI 48167

December 17, 1987

Detrex Corporation, Solvent Division
4000 Town Center, Suite 1100
Southfield, MI 48075
ATTN: Michael J. Tepatti
Project Development Manager

Dear Mr. Tepatti,

We have reviewed your transfer facility permit application submitted on November 12, 1987, for the Eaton Avenue facility. The application meets the requirements under Michigan Act 64 and the facility is approved to preceed with construction, as per Robert J. Davis (Consultant Engineer, Commission #87-1092, dated December 16, 1987) drawing #1 Sheet 1 of 1.

The actual transfer facility permit will not be issued until construction is completed and a site inspection is conducted by MDNR staff.

If you have any questions concerning this matter, please contact me at (313) 344-4670.

Sincerely,

Faye Dade

Faye Dade
Environmental Quality Analyst

FD:bs
cc: B. Okwumabua

Date: 09/03/91
Revision: 91-2

INTRODUCTION

This Resource Conservation and Recovery Act (RCRA) Part B Permit Application/Act 64 Operating License Application (hereinafter "operating license") is submitted in response to the State of Michigan, Department of Natural Resource's (MDNR's) letter of request dated March 8, 1988 and the March and August 1991, Notice of Deficiencies.

Detrex Corporation specializes in the sale of halogenated solvents and the recovery (recycling) of spent solvents. The facility operates under EPA Identification Number MID091605972. The Standard Industrial Classification (SIC) Codes for the facility are 2869 and 5161. The facility is located at 12886 Eaton Avenue in Detroit, Michigan and has operated at this location since 1966.

The hazardous waste recovery (recycling) system consists of process tanks which have been re-designated as hazardous waste storage tanks, distillation units and a generator accumulation tank. A hazardous waste container storage area is used to store wastes prior to recycling. The entire operation is located within a single enclosed building.

The recycling operation is not subject to regulation under 40 CFR Parts 264 or 270 pursuant to 40 CFR § 261.6(3)(iii). The 5,000 gallon generator accumulation tank used for temporary accumulation of still bottoms from the recycling operation does not require a permit (operating license) pursuant to 40 CFR § 262.34. As such, this operating license application is submitted for the hazardous waste storage area and the two re-designated hazardous waste storage tanks at the Eaton Avenue facility.

The following attachments provide the necessary information required.

Attachment A-1 provides the completed MDNR Operating License Application Forms 1 and 3.

Attachment A-2 provides a figure showing the container storage area and the re-designated hazardous waste storage tanks within the facility. A key explaining the tank/process unit numbering system related uses is also provided. It should be noted that the building floor and perimeter curbing in the warehouse and reclamation areas provide secondary containment for the entire system. The Transfer Facility (not part of this Application) located outside of the northeast end of the building is also indicated as is the Detroit Water and Sewerage Department Wastewater Discharge Permit discharge point (i.e. drain) within the Boiler room.

Attachment A-3 provides photographs of the facility building the hazardous waste container storage area and the hazardous waste storage tanks.

Attachment A-4 provides copies of all operating permits held by the facility.

Attachment A-5 presents a topographic map of the facility and the surrounding area. There are no outdoor hazardous waste treatment, storage, or disposal facilities, injection or withdrawal wells, or other wells, springs or surface water bodies within 1/4 mile of the facility.

Date: 06/24/91
Revision: 91-1

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SECTION A

ACT 64 OPERATING LICENSE APPLICATION

LIST OF ATTACHMENTS

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SECTION A

ACT 64 OPERATING LICENSE APPLICATION

The information in this section is supplied in accordance with Michigan Act 64 Rule 299.9508(1) which incorporates 40 CFR§270.13 by reference.

The following attachments provide the necessary information required.

Attachment A-1 provides the completed MDNR Operating License Application Forms 1 and 3.

Attachment A-2 provides a figure showing the container storage area and the re-designated hazardous waste storage tanks within the facility. A key explaining the tank/process unit numbering system related uses is also provided. It should be noted that the building floor and perimeter curbing in the warehouse and reclamation areas provide secondary containment for the entire system. The Transfer Facility located outside of the northeast end of the building is also indicated as is the Detroit Water and Sewerage Department Wastewater Discharge Permit discharge point (i.e. drain) within the Boiler room.

Attachment A-3 provides photographs of the facility building the hazardous waste container storage area and the hazardous waste storage tanks.

Attachment A-4 provides copies of all operating permits held by the facility.

Attachment A-5 presents a topographic map of the facility and the surrounding area. There are no outdoor hazardous waste treatment, storage, or disposal facilities (with the exception of the Transfer Facility), injection or withdrawal wells, or other wells, springs or surface water bodies within 1/4 mile of the facility.

ATTACHMENTS

ATTACHMENT A-1

MDNR OPERATING LICENSE APPLICATION

FORMS 1 AND 3

FORM 1 GENERAL

DNR

MICHIGAN DEPARTMENT OF NATURAL RESOURCES

(Read the "General Instructions" before starting.)

I. EPA I.D. NUMBER: F MID091605972 D

APPLICATION FOR HAZARDOUS WASTE TREATMENT, STORAGE OR DISPOSAL FACILITY CONSTRUCTION PERMIT OR OPERATING LICENSE

II. CONSTRUCTION PERMIT OR OPERATING LICENSE APPLICATION (check one)

A. CONSTRUCTION PERMIT APPLICATION

X B. OPERATING LICENSE APPLICATION

If this is an operating license application, mark an X in the appropriate box:

1. ☐ FIRST APPLICATION (NEW FACILITY)
2. ☒ FIRST APPLICATION (EXISTING FACILITY)
3. ☐ RENEWAL APPLICATION
4. ☐ APPLICATION FOR LICENSE REVISION
5. ☐ RESEARCH, DEVELOPMENT & DEMONSTRATION LICENSE APPLICATION

III. NAME OF FACILITY

1 SKIP Detrex Corporation Solvents and Environmental Services Division

IV. FACILITY CONTACT

A. NAME & TITLE (last, first, & title)	B. PHONE (area code & no.)
2 William M. Moore Jr. Corporate Manager of Environmental Compliance RCRA Section	313 358 5800

V. FACILITY MAILING ADDRESS

A. STREET OR P.O. BOX	B. CITY OR TOWN	C. STATE	D. ZIP CODE
3 O Box 5111	4 Southfield	MI	48086

VI. FACILITY LOCATION

A. STREET, ROUTE NO. OR OTHER SPECIFIC IDENTIFIER	B. COUNTY NAME
5 12886 Eaton Avenue	Wayne

C. CITY OR TOWN	D. STATE	E. ZIP CODE	F. COUNTY CODE (if known)
6 Detroit	MI	48227	

VIB. TITLEHOLDER OF LAND

A. NAME: 8 Detrex Corporation

E. STREET OR P.O. BOX: Box 5111

F. CITY OR TOWN	G. STATE	H. ZIP CODE	D. PHONE (area code & no.)
B[illegible]outhfield	MI	48086	A 313 358 5800

A. FIRST	B. SECOND
7 2869 (specify)	7 5161 (specify)

C. THIRD	D. FOURTH
7 (specify)	7 (specify)

OPERATOR INFORMATION

A. NAME: 8 Detrex Corporation Solvents and Environmental Services Division

B. Is the name listed i Item VIII-A also th owner? [X] YES [ ] NO

C. STATUS OF OPERATOR (Enter the appropriate letter into the answer box; if "Other", specify.)
F = FEDERAL
S = STATE
P = PRIVATE
M = PUBLIC (other than federal or state)
O = OTHER (specify)

P (specify)

D. PHONE (area code & no.): A 313 358 5800

E. STREET OR P.O. BOX: PO Box 5111

F. CITY OR TOWN	G. STATE	H. ZIP CODE
B Southfield	MI	48086

I. INDIAN LAND: Is the facility located on Indian lands? [ ] YES [X] NO

OWNERSHIP

A. NAME: 8 Detrex Corporation

E. STREET OR P.O. BOX: PO Box 5111

F. CITY OR TOWN	G. STATE	H. ZIP CODE	D. PHONE (area code & no.)
B Southfield	MI	48086	A 313 358 5800

EXISTING ENVIRONMENTAL PERMITS

A. NPDES (Discharges to Surface Water): 9 N

D. PSD (Air Emissions from Proposed Sources): 9 P SEE ATTATCHED

B. UIC (Underground Injection of Fluids): 9 U

E. OTHER (specify): 9 005-015 (specify) Detroit Wastewater Discharge Permit

C. RCRA (Hazardous Wastes): 9 R MID091605972

E. OTHER (specify): 9 (specify)

XI. MAP

Attach to this application a topographic map of the area extending to at least one mile beyond property bounderies. The map must show the outline of the facility, the location of each of its existing and proposed intake and discharge structures, each of its hazardous waste treatment, storage, or disposal facilities, and each well where it injects fluids underground. Include all springs, rivers and other surface water bodies in the map area. See instructions for precise requirements.

XII. NATURE OF BUSINESS (provide a brief description)

Warehouse and distribution of industrial solvents.

Reclamation of used halogenated solvents.

WAYNE COUNTY DEPARTMENT OF HEALTH
AIR POLLUTION PERMITS

APC	5-02465
APC	5-02466
APC	5-02467
APC	5-02468
APC	5-02469
APC	5-02470
APC	5-02473
APC	5-02474
APC	5-02475
APC	5-02476
APC	5-02477
APC	5-02478
APC	5-02479
APC	5-02480

Installation permit application - Dyna 500 distillation unit.

A. ☐ CONSTRUCTION PERMIT FEE

1. ☐ COST OF REVIEW

2. ☐ FIXED FEE (complete the following) — FEE

a. Check type of facility:

☐ Land Disposal ($9,000) $_____

☐ Incineration or other treatment ($7,200) $_____

☐ Storage ($500) $_____

b. Site size _____ acres (see fee schedule) $_____

c. Projected waste volume (see fee schedule)

_____ Gallons/day $_____

OR _____ Cubic yards/day $_____

d. Hydrogeological characteristics for land disposal

☐ Natural Clay

☐ Sand

☐ Compacted Clay

☐ Artificial Liner $_____

e. For treatment or storage facilies:
Is there surface water on the site?

☐ No

☐ Yes ($75) $_____

TOTAL FIXED FEE COST: $_____

B. ☒ OPERATING LICENSE FEE $ 500.00 (previously submitted

XIV. OPERATOR CERTIFICATION

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this application and all attachments and that, based on my inquiry of those persons immediately responsible for obtaining the information contained in the application, I believe that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.

A. NAME & OFFICIAL TITLE (type or print)	B. SIGNATURE	C. DATE SIGNED
Issa H. Shamiyeh - Director	X	8-28-91

XV. OWNER CERTIFICATION

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this application and all attachments and that, based on my inquiry of those persons immediately responsible for obtaining the information contained in the application, I believe that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.

A. NAME & OFFICIAL TITLE (type or print)	B. SIGNATURE	C. DATE SIGNED
Ferd J. Chmielnicki-Corp. Secretary	X Ferd J. Chmielnicki	8-28-91

XVI. TITLEHOLDER OF LAND CERTIFICATION

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this application and all attachments and that, based on my inquiry of those persons immediately responsible for obtaining the information contained in the application, I believe that the information is true, accurate and complete. I am aware that there are significant penalties for submitting f information, including the possibility of fine and imprisonment.

A E & OFFICIAL TITLE (type or print)	B. SIGNATURE	C. DATE SIGNED
Ferd J. Chmielmick-Corp. Secretary	X Ferd J. Chmielnicki	8-28-91

RCRA | DNR OF NATURAL RESOURCES | FMID091605972

FOR OFFICIAL USE ONLY

APPLICATION APPROVED	DATE RECEIVED (yr., mo., & day)	COMMENTS

FIRST OR REVISED APPLICATION

s an "X" in the appropriate box in A or B below *(mark one box only)* to indicate whether this is the first application you are submitting for your facility c sed application. If this is your first application and you already know your facility's EPA I.D. Number, or if this is a revised application, enter your facility EPA I.D. Number in Item I above.

A. FIRST APPLICATION *(place an "X" below and provide the appropriate date)*

[X] 1. EXISTING FACILITY *(See instructions for definition of "existing" facility. Complete item below.)*

[ ] 2. NEW FACILITY *(Complete item below.)*

C	YR.	MO.	DAY
8	66	06	01

FOR EXISTING FACILITIES, PROVIDE THE DATE (yr., mo., & day) OPERATION BEGAN OR THE DATE CONSTRUCTION COMMENCED *(use the boxes to the left)*

YR.	MO.	DAY

FOR NEW FACILITI PROVIDE THE DAT (yr., mo., & day) OPE TION BEGAN OR IS EXPECTED TO BEG

B. REVISED APPLICATION

[ ] 1.

[ ] 2.

III. PROCESSES – CODES AND DESIGN CAPACITIES

A. PROCESS CODE – Enter the code from the list of process codes below that best describes each process to be used at the facility. Ten lines are provided fo entering codes. If more lines are needed, enter the code(s) in the space provided. If a process will be used that is not included in the list of codes below, the describe the process *(including its design capacity)* in the space provided on the form *(Item III-C)*.

B. PROCESS DESIGN CAPACITY – For each code entered in column A enter the capacity of the process.
1. AMOUNT – Enter the amount.
2. UNIT OF MEASURE – For each amount entered in column B(1), enter the code from the list of unit measure codes below that describes the unit of measure used. Only the units of measure that are listed below should be used.

PROCESS	PROCESS CODE	APPROPRIATE UNITS OF MEASURE FOR PROCESS DESIGN CAPACITY
Storage:		
CONTAINER *(barrel, drum, etc.)*	S01	GALLONS OR LITERS
TANK	S02	GALLONS OR LITERS
WASTE PILE	S03	CUBIC YARDS OR CUBIC METERS
SURFACE IMPOUNDMENT	S04	GALLONS OR LITERS
Disposal:		
INJECTION WELL	D79	GALLONS OR LITERS
LANDFILL	D80	ACRE-FEET *(the volume that would cover one acre to a depth of one foot)* OR HECTARE-METER
ND APPLICATION	D81	ACRES OR HECTARES
EAN DISPOSAL	D82	GALLONS PER DAY OR LITERS PER DAY
SURFACE IMPOUNDMENT	D83	GALLONS OR LITERS

PROCESS	PROCESS CODE	APPROPRIATE UNITS OF MEASURE FOR PROCESS DESIGN CAPACITY
Treatment:		
TANK	T01	GALLONS PER DAY OR LITERS PER DAY
SURFACE IMPOUNDMENT	T02	GALLONS PER DAY OR LITERS PER DAY
INCINERATOR	T03	TONS PER HOUR OR METRIC TONS PER HOUR: GALLONS PER HOUR OR LITERS PER HOUR
OTHER *(Use for physical, chemical, thermal or biological treatment processes not occurring in tanks, surface impoundments or incinerators. Describe the processes in the space provided: Item III-C.)*	T04	GALLONS PER DAY OR LITERS PER DAY

UNIT OF MEASURE	UNIT OF MEASURE CODE	UNIT OF MEASURE	UNIT OF MEASURE CODE	UNIT OF MEASURE	UNIT O. MEASUR CODE
GALLONS	G	LITERS PER DAY	V	ACRE-FEET	A
LITERS	L	TONS PER HOUR	D	HECTARE-METER	F
CUBIC YARDS	Y	METRIC TONS PER HOUR	W	ACRES	B
CUBIC METERS	C	GALLONS PER HOUR	E	HECTARES	Q
GALLONS PER DAY	U	LITERS PER HOUR	H		

EXAMPLE FOR COMPLETING ITEM III *(shown in line numbers X-1 and X-2 below)*: A facility has two storage tanks, one tank can hold 200 gallons and the other can hold 400 gallons. The facility also has an incinerator that can burn up to 20 gallons per hour.

C DUP | T/A/C | I

LINE NUMBER	A. PROCESS CODE (from list above)	B. PROCESS DESIGN CAPACITY 1. AMOUNT (specify)	2. UNIT OF MEASURE (enter code)	FOR OFFICIAL USE ONLY	LINE NUMBER	A. PROCESS CODE (from list above)	B. PROCESS DESIGN CAPACITY 1. AMOUNT	2. UNIT OF MEASURE (enter code)	FOR OFFICI USE ONLY
X-1	S02	600	G		5				
X-2	T03	20	E		6				
1	S01	8 250	G		7				
2	S02	5 500	G		8				
3					9				
					10				

CONTINUE ON REVER

C. SPACE FOR ADDITIONAL PROCESS CODES OR FOR DESCRIBING OTHER PROCESSES *(code "T04")*. FOR EACH PROCESS ENTERED HERE INCLUDE DESIGN CAPACITY.

IV. DESCRIPTION OF HAZARDOUS WASTES

A. **EPA HAZARDOUS WASTE NUMBER** — Enter the four—digit number from 40 CFR, Subpart D for each listed hazardous waste you will handle. handle hazardous wastes which are not listed in 40 CFR, Subpart D, enter the four—digit number*(s)* from 40 CFR, Subpart C that describes the charac tics and/or the toxic contaminants of those hazardous wastes.

B. **ESTIMATED ANNUAL QUANTITY** — For each listed waste entered in column A estimate the quantity of that waste that will be handled on an a basis. For each characteristic or toxic contaminant entered in column A estimate the total annual quantity of all the non—listed waste*(s)* that will be ha which possess that characteristic or contaminant.

C. **UNIT OF MEASURE** — For each quantity entered in column B enter the unit of measure code. Units of measure which must be used and the appro codes are:

ENGLISH UNIT OF MEASURE	CODE	METRIC UNIT OF MEASURE	CODE
POUNDS	P	KILOGRAMS	K
TONS	T	METRIC TONS	M

If facility records use any other unit of measure for quantity, the units of measure must be converted into one of the required units of measure takin account the appropriate density or specific gravity of the waste.

D. **PROCESSES**

1. **PROCESS CODES:**
   For listed hazardous wastes: For each listed hazardous waste entered in column A select the code*(s)* from the list of process codes contained in Ite to indicate how the waste will be stored, treated, and/or disposed of at the facility.
   For non—listed hazardous wastes: For each characteristic or toxic contaminant entered in column A, select the code*(s)* from the list of process contained in Item III to indicate all the processes that will be used to store, treat, and/or dispose of all the non—listed hazardous wastes that p that characteristic or toxic contaminant.
   Note: Four spaces are provided for entering process codes. If more are needed: (1) Enter the first three as described above; (2) Enter "000" extreme right box of Item IV-D(1); and (3) Enter in the space provided on page 4, the line number and the additional code*(s)*.

2. **PROCESS DESCRIPTION:** If a code is not listed for a process that will be used, describe the process in the space provided on the form.

NOTE: HAZARDOUS WASTES DESCRIBED BY MORE THAN ONE EPA HAZARDOUS WASTE NUMBER — Hazardous wastes that can be describ more than one EPA Hazardous Waste Number shall be described on the form as follows:

1. Select one of the EPA Hazardous Waste Numbers and enter it in column A. On the same line complete columns B,C, and D by estimating the total a quantity of the waste and describing all the processes to be used to treat, store, and/or dispose of the waste.
2. In column A of the next line enter the other EPA Hazardous Waste Number that can be used to describe the waste. In column D(2) on that line "Included with above" and make no other entries on that line.
3. Repeat step 2 for each other EPA Hazardous Waste Number that can be used to describe the hazardous waste.

EXAMPLE FOR COMPLETING ITEM IV *(shown in line numbers X-1, X-2, X-3, and X-4 below)* — A facility will treat and dispose of an estimated 900 p per year of chrome shavings from leather tanning and finishing operation. In addition, the facility will treat and dispose of three non—listed wastes. Two are corrosive only and there will be an estimated 200 pounds per year of each waste. The other waste is corrosive and ignitable and there will be an estir 100 pounds per year of that waste. Treatment will be in an incinerator and disposal will be in a landfill.

LINE NO.	A. EPA HAZARD. WASTE NO. (enter code)	B. ESTIMATED ANNUAL QUANTITY OF WASTE	C. UNIT OF MEASURE (enter code)	D. PROCESSES 1. PROCESS CODES (enter)	D. PROCESSES 2. PROCESS DESCRIPTION (if a code is not entered in D(1))
X-1	K 0 5 4	900	P	T 0 3 D 8 0	
X-2	D 0 [illegible] 2	400	P	T 0 3 D 8 0	
X-3	D 0 0 1	100	P	T 0 3 D 8 0	
X-4	D 0 0 2				*included with above*

EPA I.D. NUMBER (enter from page 1)	FOR OFFICIAL USE ONLY
W M I D 0 9 1 6 0 5 9 7 2 . 1	W DUP 2 DUP

IV. DESCRIPTION OF HAZARDOUS WASTES (continued)

W	A. EPA HAZARD. WASTE NO. (enter code)	B. ESTIMATED ANNUAL QUANTITY OF WASTE	C. UNIT OF MEASURE (enter code)	D. PROCESSES 1. PROCESS CODES (enter)	D. PROCESSES 2. PROCESS DESCRIPTION (if a code is not entered in D(1))
1	F001	3 795 000	P	S01 S02	
2	F002	250 000	P	S01 S02	
3	D004	Note: The waste	P	S01 S02	
4	D005	Quantities reported	P	S01 S02	
5	D006	For F001 and F002 include the	P	S01 S02	
6	D007	D-Series compounds	P	S01 S02	
7	D008		P	S01 S02	
8	D009		P	S01 S02	
9	D010		P	S01 S02	
10	D011		P	S01 S02	
11	D018		P	S01 S02	
12	D019		P	S01 S02	
	D021		P	S01 S02	
14	D022		P	S01 S02	
15	D027		P	S01 S02	
16	D028		P	S01 S02	
17	D029		P	S01 S02	
18	D030		P	S01 S02	
19	D034		P	S01 S02	
20	D035		P	S01 S02	
21	D036		P	S01 S02	
22	D038		P	S01 S02	
23	D039		P	S01 S02	
24	D040		P	S01 S02	
25	D043		P	S01 S02	
?					

CONTINUE ON REVERSE

E. USE THIS SPACE TO LIST ADDITIONAL PROCESS CODES FROM ITEM D(1) ON PAGE 3.

EPA I.D. NO. (enter from page 1)

F	M	I	D	0	9	1	6	0	5	9	7	2	6

V. FACILITY DRAWING

All existing facilities must include in the space provided on page 5 a scale drawing of the facility *(see instructions for more detail).*

VI. PHOTOGRAPHS

All existing facilities must include photographs *(aerial or ground—level)* that clearly delineate all existing structures; existing storage, treatment and disposal areas; and sites of future storage, treatment or disposal areas *(see instructions for more detail).*

VII. FACILITY GEOGRAPHIC LOCATION

LATITUDE (degrees, minutes, & seconds)	LONGITUDE (degrees, minutes, & seconds)
42 23 50	83 10 22

VIII. GENERAL INFORMATION

Attach each of the following as separate attachments to the application:

1. General facility description
2. Chemical & physical analysis
3. Waste analysis plan
4. Security procedures
5. Inspection schedule
6. Contingency plan
7. Preparedness/prevention
8. Traffic information
9. Location information
10. Training program
11. Closure/post-closure pl
12. Cost estimates
13. Liability mechanism
14. Financial assurance
15. Topographic map

IX. SUPPLEMENTAL INFORMATION

Attach for all applications:

1. Hydrogeological report
2. Environmental assessment
3. Environmental monitoring program
4. Engineering plans

Attach for operating license applications only

1. For new facilities, construction certificat
2. Capability certification/compliance schedul
3. Proof of other permits or licenses
4. Restrictive covenant (landfills only)

X. FACILITY SPECIFIC INFORMATION

Attach the required technical information for each of the following:

1. Containers
2. Tanks
3. Incineration or thermal treatment
4. Treatment
5. Surface impoundments
6. Waste piles
7. Landfills
8. Land treatment

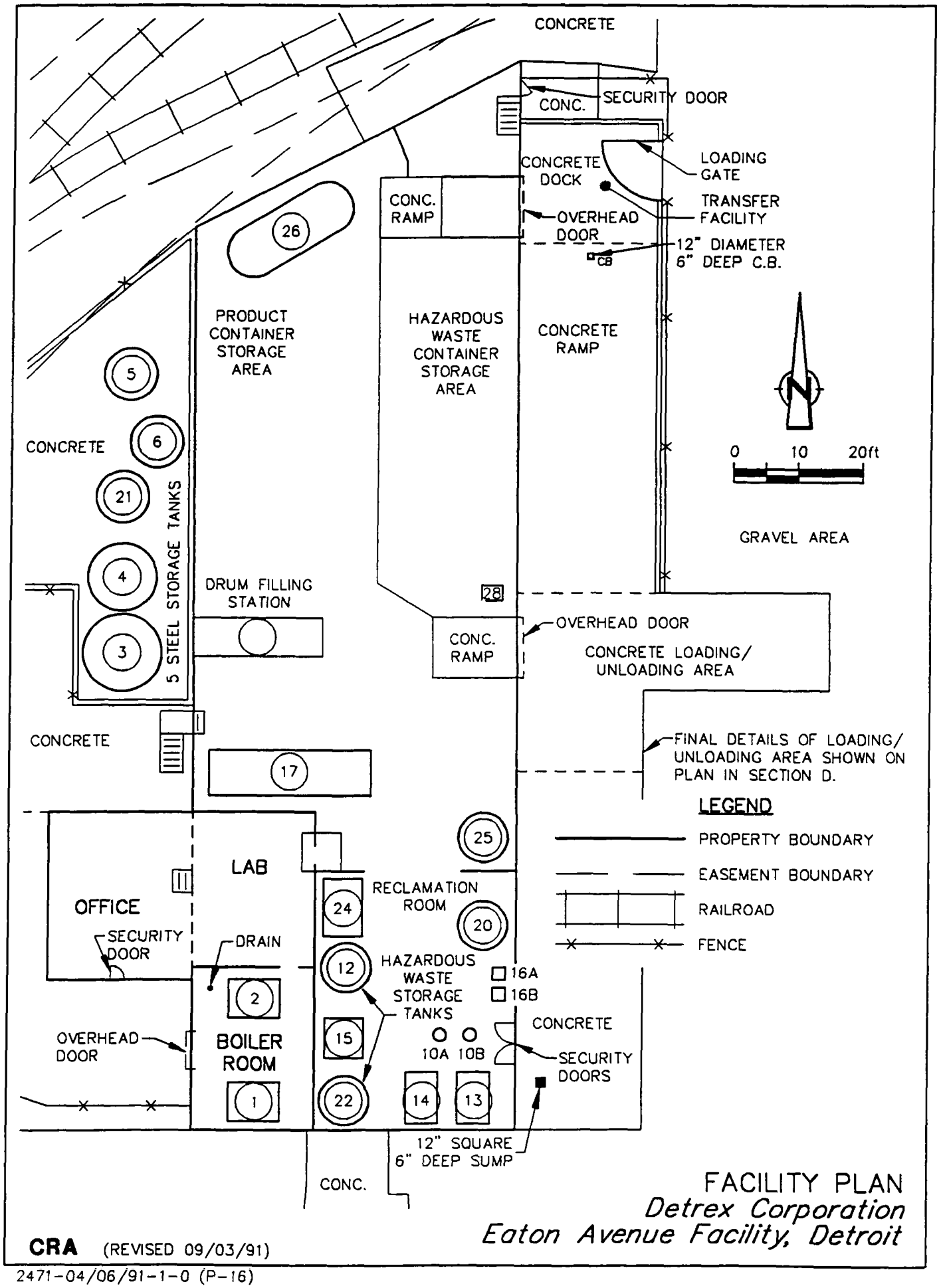
CONTINUE ON

Note: See attatchment A-2

ATTACHMENT A-2

FACILITY PLAN

ATTACHMENT A-2



FACILITY PLAN
Detrex Corporation
Eaton Avenue Facility, Detroit

CRA (REVISED 09/03/91)

2471-04/06/91-1-0 (P-16)

WAYNE COUNTY DEPARTMENT OF HEALTH

AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502513)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02465

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 TRI STORAGE TANK #1

SOURCE ID: 003 CAPACITY: 20,000 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

THIS CERTIFICATE ISSUED BY
DIVISION OF AIR POLLUTION CONTROL
ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

THIS CERTIFICATE IS SUBJECT TO SPECIAL OPERATING CONDITIONS IF NOTED ON THE REVERSE SIDE OF THIS FORM AND/OR AS STATED ON THE INSTALLATION PERMIT ISSUED BY THE DIVISION FOR THIS EQUIPMENT.

APC-23 (REV 3-84)

ATTACHMENT A-3

FACILITY PHOTOGRAPHS

① TRANSFER FACILITY

② LOADING/UNLOADING AREA – MIDDLE OVERHEAD DOOR ON EAST SIDE OF FACILITY

③ F001 / F002 HAZARDOUS WASTE STORAGE TANK

④ STILL BOTTOMS STORAGE TANK

CRA

PHOTOGRAPHIC LOG
Detrex Corporation
Eaton Avenue Facility, Detroit

2471-13/06/91-1-0

(5) INTERIOR VIEW OF BUILDING FROM NORTH END (PROCESS AREA)

(6) FORKLIFT TRUCK EQUIPPED WITH DRUM HANDLING ATTACHMENT, (PRODUCT DRUM STORAGE ON WEST SIDE)

(7) HAZARDOUS WASTE CONTAINER STORAGE AREA

(8) MIDDLE OVERHEAD DOOR ON EAST SIDE OF FACILITY, (ENTRANCE FROM LOAD/UNLOAD AREA) RAMP PROVIDES SECONDARY CONTAINMENT

ATTACHMENT A-4

PERMITS

WAYNE COUNTY DEPARTMENT OF HEALTH
AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502514)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02466

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 I.I.I. STORAGE TANK #2

SOURCE ID: 004 CAPACITY: 16,000 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH

AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502515)	13/1442 ESTABLISHMENT NO B2273	APC 5-02467

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 STORAGE TANK RECLAIMED TRI #4

SOURCE ID: 004 CAPACITY: 5,000 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH

AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502516)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02468

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 PERK STORAGE TANK #5

SOURCE ID: 005 CAPACITY: 5,000 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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DIVISION OF AIR POLLUTION CONTROL
ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH
AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502517)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02469

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 METHYLENE STORAGE TANK #3

SOURCE ID: 006 CAPACITY: 5,000 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY L.. ARTMENT OF HEALTH
AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA GRID SQUARE	CERTIFICATE OF OPERATION
12806 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502518)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02470

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 RECLAIMED TRI TANK F002B

SOURCE ID: 007 CAPACITY: 1500.00 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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DIVISION OF AIR POLLUTION CONTROL
ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH

AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502521)	13/1442	APC 5-02473
	ESTABLISHMENT NO. B2273	

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 RECEIVER TANK AQS

SOURCE ID: 010 CAPACITY: 750.00 GALS.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH
AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502522)	13/1442	APC 5-02474
	ESTABLISHMENT NO B2273	

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 0086 STILL FEED TANKS-FOO 1 TWO UNITS

SOURCE ID: 012 CAPACITY 2,000 GAL. TANK - 4,000 GAL. TANK

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ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH
AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12836 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502523)	13/1442	APC 5-02475
	ESTABLISHMENT NO D2273	

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 0085 S-350 STILL DETREX
CONTROL EQUIP: TOTAL CONDENSERS
SOURCE ID: 013 CAPACITY: 300.00 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH

AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502524)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02476

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 0085 S-600 STILL AOM
CONTROL EQUIP: TOTAL CONDENSERS
SOURCE ID: 014 CAPACITY: 400.00 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

THIS CERTIFICATE ISSUED BY
DIVISION OF AIR POLLUTION CONTROL
ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH
AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502525)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02477

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 0085 DCI STILL 551-16-02 (TWO)
CONTROL EQUIP: TOTAL CONDENSERS
SOURCE ID: 015 CAPACITY: ONE 60 GAL TANK; ONE 500 GAL TANK

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

THIS CERTIFICATE ISSUED BY
DIVISION OF AIR POLLUTION CONTROL
ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH

AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502526)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02478

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 DRYING COLUMN (TWO)

SOURCE ID: 016

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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DIVISION OF AIR POLLUTION CONTROL
ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87

Air Pollution Inspector — Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH

AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502527)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02479

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 0086 BOTTOMS TANK FOO 2

SOURCE ID: 017 CAPACITY: 5000.00 GAL.

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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DIVISION OF AIR POLLUTION CONTROL
ALAN M. GREENBERG, *Director*

Lee Murchison 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

WAYNE COUNTY DEPARTMENT OF HEALTH
AIR POLLUTION CONTROL DIVISION
2211 E. JEFFERSON, DETROIT, MICHIGAN 48207
TELEPHONE NO. 567-1180

LOCATION	AREA/GRID SQUARE	CERTIFICATE OF OPERATION
12886 EATON AVENUE DETROIT, MI. 48227 GOLD SHIELD SOLVENT DIV'N. (502528)	13/1442 ESTABLISHMENT NO. B2273	APC 5-02480

CERTIFICATE OF OPERATION

OWNER/AGENT

MICHAEL J. TEPATTI

EQUIPMENT

GENERAL PROCESS: 9999 DRUMMING STATION

SOURCE ID: 018

The possession of this certificate of operation shall not exempt any person from citation for operating the above described equipment in violation of the Wayne County Air Pollution Control Ordinance. This certificate of operation shall be posted on or near the installation described. This certification of operation may be revoked for cause.

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ALAN M. GREENBERG, *Director*

Lee Munchson 7-13-87
Air Pollution Inspector Date

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APC-23 (REV 3-84)

LOCATION OF INSTALLATION (Street address and city) (Please Print)

AREA/GRID

PERMIT NO.

For Office use only

Establishment

Source Id. No.

Equip. Code No.

Cashier

WAYNE COUNTY HEALTH DEPARTMENT
Air Pollution Control Division
2211 E. Jefferson Avenue
Detroit, Michigan 48207
Telephone No. (313) 567-0710

INSTALLATION PERMIT

Owner Gold Shield Solvents Division of Detrex Corporation Telephone No. (313) 491-4550

Mailing Address 12886 Eaton Detroit MI 48227

Installer Detroit Equipment Installation Co. Telephone No. (313) 682-4506

Address 2900 Orchard Lake Rd., Keego Harbor, MI 48033

(Circle One) To (Install) (Construct) (Reconstruct) (Relocate) (Alter) (Modify the Use of A Process)

Designate whether this application is for process equipment or emission control equipment by circling (1) or (2) below.

1. Description of Process Equipment Dyna-500 Distillation Unit (e.g. Boiler, Incinerator, Other)
   Name of Manufacturer DCT Corporation Model No. Dyna-500 Rating 500 Gal./hr.
2. Description of Emission Control Equipment Atmospheric Condenser (Total)
   Name of Manufacturer Perfex Inc. Model No. C-620 Rating 657 s.f.
3. (New, (Existing) Stack or Vent I.D. or Size 2" FNPT Height (ground to top) 5 ft
4. Signature of Owner or Agent Ronald E. Swan Jr. Title Operations Manager
5. Print Name of Owner or Agent Ronald E. Swan, Jr.

DO NOT WRITE BELOW THIS LINE

Approval by _____ / _____ with conditions in letter dated _____
Director Date

FEE BASED ON _____ EXAMINATION FEE: $ _____ INSPECTION FEE: $ _____

THIS IS NOT A CERTIFICATE OF OPERATION

APPLICANT'S APPROVED I.P.

APC-22A SEE REVERSE OF GREEN COPY FOR APPLICATION REQUIREMENTS Revised 1987

Detroit Water and Sewerage Department
Water Board Building
Detroit, Michigan 48226
(313) 224-4800

Coleman A. Young, Mayor
City of Detroit

DETROIT

WATER AND SEWERAGE DEPARTMENT

WASTEWATER DISCHARGE PERMIT - TYPE 3

Section A: General Information Permit No. 005-015

Company Name: Gold Shield Solvents Div.; Detrex Chemical Inds., Inc.

Premise Address: 12886 Eaton Ave.

City Detroit, MI Zip Code 48227

Mailing Address P. O. Box 501

City Detroit, MI Zip Code 48232

Primary Standard Industrial Classification (SIC) Code 5161

Other SIC Codes

The above Industrial User is authorized to discharge industrial wastewater to the City of Detroit sewer system in compliance with the City's Wastewater Discharge Ordinance and any applicable provisions of federal or state law or regulation, and in accordance with discharge point(s), effluent limitations, monitoring requirements, and other conditions set forth herein.

This permit is granted in accordance with the application filed in the office of Director of DWSD, and in conformity with plans, specifications and other data submitted to the City in support of the above application.

Effective Date: January 4, 1988

Expiration Date: March 1, 1992

Charlie J. Williams
Director

Section B: Discharge Limitations and Monitoring Requirements

Representative Sampling Location: At a drain: 1' E from W end of Boiler Room,

( ) (See attached schematic) 1' S from n end of Boiler Room.

The following pretreatment standards shall be applied to this location.

Parameter	Daily Maximum (mg/l) A*	Self-Monitoring Required (Y/N)
Total Arsenic (AS)	1.0	Y
Total Cadmium (Cd)	2.0	Y
Total Copper (Cu)	4.5	Y
Total Cyanide (CN)	2.0	Y
Total Lead (Pb)	1.0	Y
Total Mercury	0.005	Y
Total Nickel (Ni)	5.0	Y
Total Silver (Ag)	2.0	Y
Total Chromium (Cr)	25.0	Y
Total Zinc (Zn)	15.0	Y
PCB - Acolor 1260	0.0005	N
Total PCB	0.001	N
Fats, Oils, Grease (FOG)	2000	Y
Total Suspended Solids (TSS)	10000	Y
Biochemical Oxygen Demand (BOD)	10000	Y
Phosphorus (P)	500	Y

Other Requirements:

(1) Compliance with the General Pollutant Prohibitions

(2) pH between 5.0 - 10.0 (10.5 if alkalinity is less than 300 ppm)

* A - All limitations based on composite samples.

Section C: Self-Monitoring Requirements

All samples shall be representative of the daily operations of the facility. Sampling shall be composite sampling, unless adequate demonstration is made to the Board for other sampling. Samples must be obtained from the sampling location(s) indicated in Section B.

Sampling Location No.	Frequency of Sampling	Pollutants to be Analyzed
#1 (Page 2)	1 Day 24 hr. Composite Sample taken once every 6 months	As, Cd, Cu, CN, Pb, Hg, Ni, Ag, Cr, Zn, BOD, FOG, TSS, P

Section D: Compliance Requirements

1) Compliance Requirements (X) Not Applicable

The permittee has a compliance agreement with the Detroit Water and Sewerage Department, see Attachment _____.

2) Spill Prevention Plan (SPP) ( ) Not Applicable

The permittee is required to submit a Spill Prevention Plan in accordance with the Detroit guidelines, to provide protection against accidental discharges to POTW.

(X) The SPP has been submitted to DWSD.

( ) The SPP plan shall be submitted by _____.

3) Reporting Requirements

A Six Month Self-Monitoring Report (SMR) shall be submitted to the Department on June 30 and December 31 of each year.

The report shall be signed, dated, and certified by an authorized representative of the Industrial User, or a registered professional engineer who is knowledgeable of the facility's discharge.

This report shall include:

(i) Wastewater Analyses (see Section C)

(ii) Report whether the applicable pretreatment standards are being met on a consistent basis and, if not, what additional operation and maintenance practices and/or pretreatment construction is necessary to bring the Industrial User into compliance.

The report must include a schedule, and specify interim and final dates to achieve compliance.

(iii) The following additional requirement. ( ) Not applicable

GENERAL TERMS AND CONDITIONS

The permittee shall apply the discharge limits specified in Section B of this permit and shall comply with all provisions of the City of Detroit Wastewater Discharge Control Ordinance, other applicable laws, rules, regulations, user charges and fees established by the City of Detroit without repetition herein.

1) Records for monitoring activities shall be maintained in accordance with the ordinance requirements, and shall include for all samples:

   a) The date, exact place, time and method of sampling

   b) The names of persons taking the sample

   c) The technique or method of analysis, the date and results of analysis

   d) The names of persons performing the analysis

2) Notification

   a) Accidental Discharge

   Within one (1) hour of becoming aware of an accidental discharge into the sewer system which exceeds or does not conform with federal, state or Detroit laws, rules, regulations or permit requirements and the degree of exceedance appears to be more than twice the allowable concentration, the permittee shall telephone the Department at its control centers as shown below. Within five (5) calendar days after the discharge, the permittee shall submit a detailed written report describing the cause of the discharge and the measures to be taken by the permittee to prevent similar future occurrences.

   b) Upsets

   The permittee shall submit information on the upset orally or in writing, within 24 hours of becoming aware of the upset. If information is provided orally, a written submission must be provided within five (5) days.

   Control Centers

- Systems Control Center	224-4775
- Wastewater Treatment Plant	297-9000
- Industrial Waste Control Office	935-4444

3) Limitations on Permit Transfer

The wastewater discharge permit shall not be reassigned or transferred without the written approval of the City of Detroit Water and Sewerage Department. The permittee shall notify the Department of any such changes thirty (30) days prior to the change.

4) Modifications or Revisions of the Permit

The permittee should notify the Department of any facility or operational changes which may effect the permit.

The terms and conditions of the permit may be subject to modification by the City of Detroit Water and Sewerage Department during the term of the permit, in accordance with the City's ordinance.

5) Confidential Information

Except for data determined to be confidential under the City's ordinance, all information and data on the permittee obtained from written reports, questionnaires, permit applications, permits, monitoring programs and inspections shall be available to the public or other government agencies without restriction.

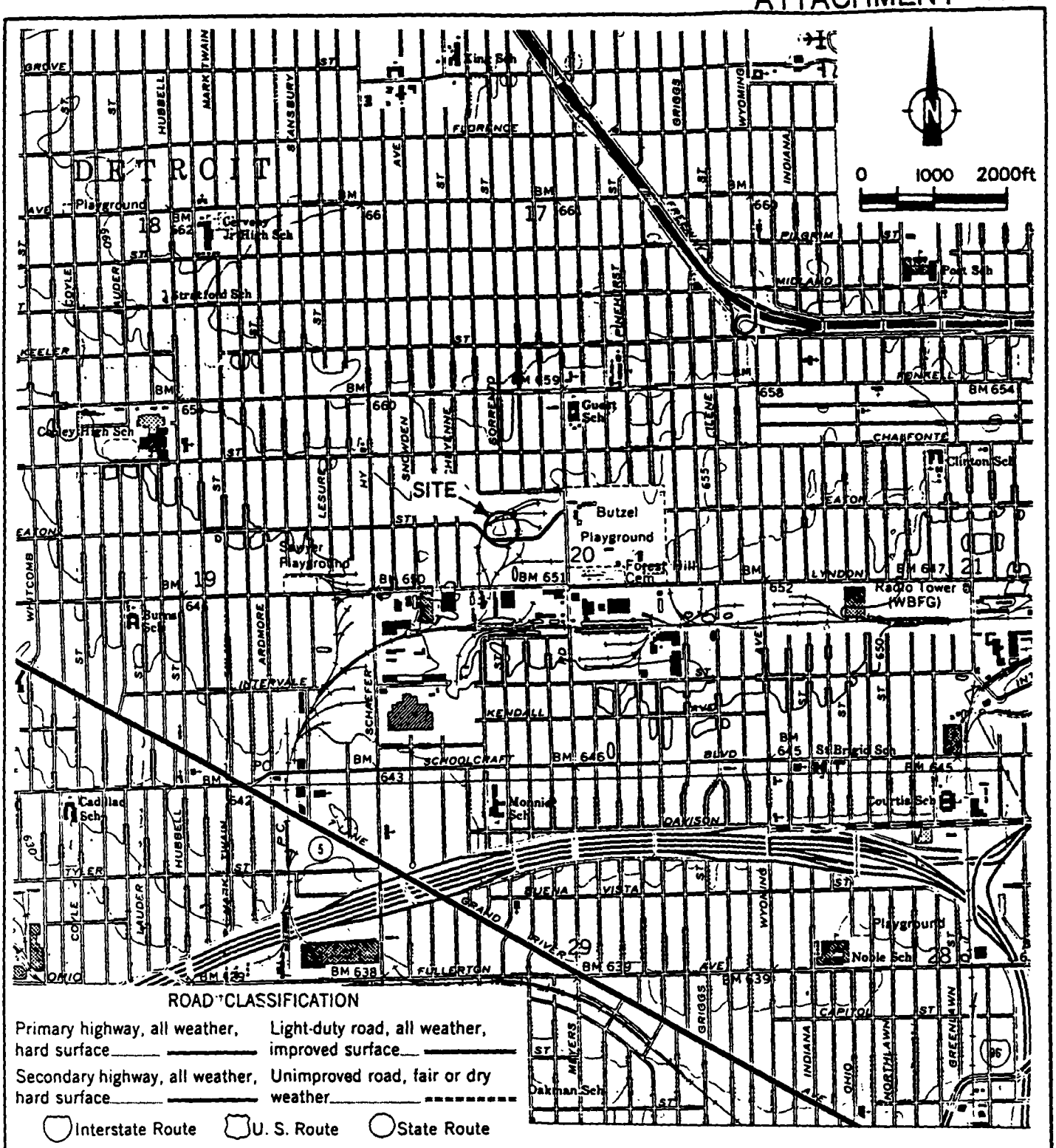
6) All reports shall be addressed to:

Detroit Water and Sewerage Department
735 Randolph Street
Detroit, Michigan 48226

Attention: Industrial Waste Control Division

ATTACHMENT A-5

TOPOGRAPHIC MAP



ROYAL OAK, MICH.
N4222.5—W8307.5/7.5

1968
PHOTOREVISED 1981
DMA 4368 I NW—SERIES V862

SURROUNDING AREA TOPOGRAPHIC MAP

Detrex Corporation
Eaton Avenue Facility, Detroit

CRA

2471-28/07/88-1-D-1

SECTION B

GENERAL DESCRIPTION

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SECTION B

FACILITY DESCRIPTION

This section of the operating license application provides a general description of the Detrex Corporation facility in Detroit, Michigan as required by Michigan Act 64 Rule 299.9504(1)(c) which incorporates 40 CFR 270.14(b) and (c) by reference. The applicable section(s) of the federal regulations (40 CFR) is referenced where appropriate.

B-1 FACILITY DESCRIPTION [40 CFR §270.14(b)(1)]

This Operating License Application is submitted by Detrex Corporation, for the hazardous waste container and tank storage facility located on Eaton Avenue in Detroit, Michigan. The facility is a treatment, storage, disposal (TSD) facility, by definition, for halogenated hydrocarbon solvents and spent solvents.

The facility is located in Wayne County, Detroit, Michigan. The Street address is:

12886 Eaton Avenue
Detroit, Michigan 48227
Facility Manager: Mr. Ronald Hritzkowin
Phone Number: 313-491-4550

The principal contact person for this Application is:

Mr. Bill Moore
Corporate Manager of Environmental Compliance, RCRA Section
P.O. Box 5111
Southfield, Michigan 48086-5111
Phone Number: 313-358-5800

The Eaton Avenue facility specializes in the sale of halogenated solvents and the recovery (recycling) of spent solvents. Spent solvents are collected from such industries as: the metal working industries that clean machined and stamped parts in vapor degreasing equipment; rubber moulding operations where defective parts are solvent removed from metal inserts; the electronic industry where circuit boards and other components are cleaned and defluxed in batch and continuous conveyor type solvent defluxing; and industries that spray paint on an assembly line basis where the hangers and conveyor components are cleaned with solvents in on-line vapor degreasers. The facility operates under EPA Identification Number MID091605972. The Standard Industrial Classification (SIC) Codes for the facility are 2869 and 5161.

The hazardous waste management operations include container storage and tank storage. Wastes are received in container or bulk shipments. The wastes are processed through the recovery (recycling) system which primarily consists of distillation followed by drying. The recovered solvent product is drummed or stored in product tanks for re-sale and the still bottoms remaining after distillation are transferred to a 5,000 gallon

generator accumulation tank. The still bottoms are accumulated for less than 90 days prior to shipment off site to a permitted facility. The entire operation is located within a single enclosed building.

All wastes accepted at the facility and the still bottoms remaining after the recovery (recycling) operation are classified as F001 or F002 hazardous wastes under 40 CFR Part 261. The wastes, under the TCLP rules, are also recognized as containing D-series compounds.

B-2 TOPOGRAPHIC MAP [40 CFR §270.14(b)(19)]

B-2a General Requirements [40 CFR §270.14(b)]

The topographic mapping and other location information requirements are listed on Table B-1 with the location in this operating license application where the appropriate map/information is located. Provided as Attachment B-1 is a figure that locates Detroit within the State of Michigan and the Eaton Avenue facility within the City of Detroit.

1) Topographic Map

Attachment B-2 provides a topographic map, at a scale of 1"=20', of the Detrex property. The topography of the surrounding area is illustrated on a portion of the United States Department of the Interior Geological Survey Quadrangle Map included as Attachment B-3.

2) Land Use

Attachments B-4 and B-5 provide maps of the zoning and existing land-use, respectively, in the vicinity of the facility. The zoning code for the industrial area in which the Detrex property is located is M4 while the surrounding area is R1. The M4 zoning code indicates an intensive industrial district while R1 indicates a single-family residential district. The closest residential property is over 300 feet west of the facility.

The figures were reproduced from figures supplied by the Planning Department of the City of Detroit.

3) Wind Rose

A wind rose for the Detroit City Airport is presented in Attachment B-6. The airport is located approximately 35 miles east of the facility.

4) Legal Boundaries

A copy of a legal survey indicating the legal boundaries and a legal description of the lands owned by Detrex Corporation is provided as Attachment B-7.

TABLE B-1

LOCATION IN APPLICATION OF TOPOGRAPHIC MAP REQUIREMENTS

Items Required By 40 CFR §270.14(b)(19) For Topographic Maps		*Location In Application (Attachment Nos.)**
1.	Topographic map	B-2, B-3
2.	Contour intervals	B-2, B-3
3.	Map scale and date	B-2, B-3, B-5, B-7, B-10
4.	Orientation of map (i.e. North arrow)	B-1, B-2, B-3, B-5, B-7, B-8, B-9, B-10
5.	Legal boundaries (i.e. property line)	B-2, B-7
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9.	Hazardous waste container storage area	B-9
10.	100-year floodplain area	B-10
11.	Location of access control (i.e. fences gates)	B-8
12.	Access and internal roads	B-8
13.	Drainage barriers	B-2
14.	Run-off control systems	B-2
15.	Buildings and structures	B-2, B-7, B-8, B-9
16.	Sewers (i.e. process, sanitary, storm)	B-2, B-7
17.	Loading/unloading areas	B-2, B-7, B-8, B-9
18.	Fire control facilities	B-2, B-7
19.	Withdrawal wells	N/A
20.	Injection wells	N/A
21.	Groundwater information (i.e. point of compliance, monitoring wells, groundwater flow direction and rate)	B-2

* N/A - Not Applicable

5) Access Control

Attachment B-8 illustrates the access control devices at the facility. The hazardous waste storage areas are enclosed within a single building provided with security doors that remain locked when the facility is unattended. In addition, much of the facility is surrounded by an 8-foot high chain link fence that is topped by razor wire. Entrance through the fence is controlled by gates that remain locked at all times.

6) Injection and Withdrawal Wells

There are no available records of any groundwater injection or withdrawal wells within a one mile radius of the Eaton Avenue facility.

7) Sewers

The location of sewers and other utilities is provided on the legal survey plan located in Attachment B-7. The drain in the boiler room is a discharge point as permitted under a Detroit Water and Sewerage Industrial Wastewater Discharge Permit.

8) Loading and Unloading Areas

The loading and unloading area associated with the hazardous waste areas at the facility is shown in Attachment B-8. The internal access roads are also shown.

9) Fire Control

There are two fire hydrants, serviced by an 8-inch diameter watermain, located near the facility along Eaton Avenue. The nearest is only about 25 feet south of the south end of the facility. These fire hydrants are shown on the legal plan located in Attachment B-7.

10) Flood Control

As indicated in Section B-3b of this operating license application, the Detrex facility is not located in an area designated as a 100-year floodplain. As such, flood control structure/devices are not required. Not withstanding the above, flood prevention is provided by the enclosed building structure that houses the hazardous waste container and tank storage areas and the recycling operation process equipment.

The outdoor loading/unloading area secondary containment system (i.e. perimeter curbing and drive-over access ramp) has been designed to accommodate a 24-hour 100-year rainfall event in addition to the maximum bulk waste shipment.

11) Runoff Control

The topographic maps provided in Attachments B-2 and B-3 indicate the surface water drainage pattern around the facility. Surface water runoff is directed away from the building structure in all areas except along a portion of the east wall. A 12-inch square, 6-inch deep sump is located within the concrete loading/unloading area to provide runoff control. The accumulated runoff collected is tested in accordance with a Detroit Water and Sewerage Department Industrial Wastewater Discharge Permit. If the water meets the conditions of the permit, it is discharged into the municipal sanitary sewer system through a drain located in the Boiler Room. If the water fails to meet the permit requirements for direct discharge, it will be collected and containerized for off-Site disposal in accordance with Federal, State and Local regulations. A 12-inch diameter, 6-inch deep catchbasin is located within the Transfer Facility, however this is independent of the TSD operation and no accumulated liquids are discharged to the ground.

As indicated in Attachment B-3, the area of Detroit in which the facility is located, slopes gradually to the south towards the Detroit River.

12) Hazardous Waste Container Storage
Area and Hazardous Waste Storage Tanks

Provided in Attachment B-9 is a figure detailing the location of the hazardous waste management units within the building.

B-2b Additional Requirements for Land Disposal Facilities
[40 CFR §270.14(c)(3),§270.25(c)(4),§264.95 and §264.97]

Detrex Corporation does not and has never had a hazardous waste land disposal facility at the Eaton Avenue facility in Detroit, Michigan.

B-3 FACILITY LOCATION [40 CFR §270.14(b)(11)]

B-3a Seismic Standard

The Detrex Corporation facility is located in Wayne County, Detroit, Michigan. It is located at latitude N42°-23'-50" and longitude W83°-10'-22".

The seismic standard is not applicable to facilities located in Michigan.

B-3b Floodplain Standard
[40 CFR §270.14(b)(11)(iii), §264.18(b)]

Attachment B-10 shows the portion of the Federal Insurance Administration (FIA) flood map for the City of Detroit relative to the facility. The FIA map indicates that the facility is in neither the 100-year nor the 500-year floodplain. This area is entirely within Zone C indicating it is not subject to flooding.

The facility is located approximately 7.5 miles northwest of the Detroit River and 5 miles east of the River Rouge. The 100-year flood elevation of the Detroit River is approximately 578 feet above National Geodetic Vertical Datum (NGVD). The facility property elevation is approximately 655 feet above NGVD.

B-4 TRAFFIC INFORMATION [40 CFR §270.14(b)(10)]

Attachment B-8 illustrates the traffic patterns at the Eaton Avenue facility. Vehicular access to the facility is direct via Eaton Avenue from Schaefer Avenue to the west or Meyers Road to the east. The facility is serviced primarily by Highway 10 and Interstate 96, both major freeways in the area. Visitors and employees park directly in front of the building or inside the fenced enclosure to the west of the building.

All traffic associated with the movement of hazardous waste occurs to the east of the building. All incoming hazardous wastes arrive by truck. Transport of the waste is made using one of the four types of Detrex owned licensed hazardous waste transporter vehicles, incuding: 1) stake trailer, 2) tank trailer, 3) vacuum tank trailer and 4) vacuum pumper, as required and according to Federal and State laws.

Bulk waste is pumped directly into hazardous waste storage tanks from the delivery trucks via a system of vacuum tubing with quick release couplings installed within the facility. The bulk shipment vehicles park on the loading/unloading dock located on the east side of the building. All material transfer is conducted under procedures designed to minimize the risk of a possible spill of the hazardous waste.

A forklift truck equipped with a special drum handling attachment is used to transfer drummed waste into and within the building.

Hazardous wastes generated from the recycling operation are transported off site by tanker truck Wastes are transported off site at a frequency of approximately two tanker trucks per month.

All paved roads at the facility are constructed of a minimum of six inches of concrete. Historical traffic flow at the facility has not created any structural problems in the existing roads on site, indicating the bearing capacity of the roads is adequate.

ATTACHMENTS

ATTACHMENT B-1

LOCATION PLAN

SITE LOCATION
Detrex Corporation
Eaton Avenue Facility, Detroit

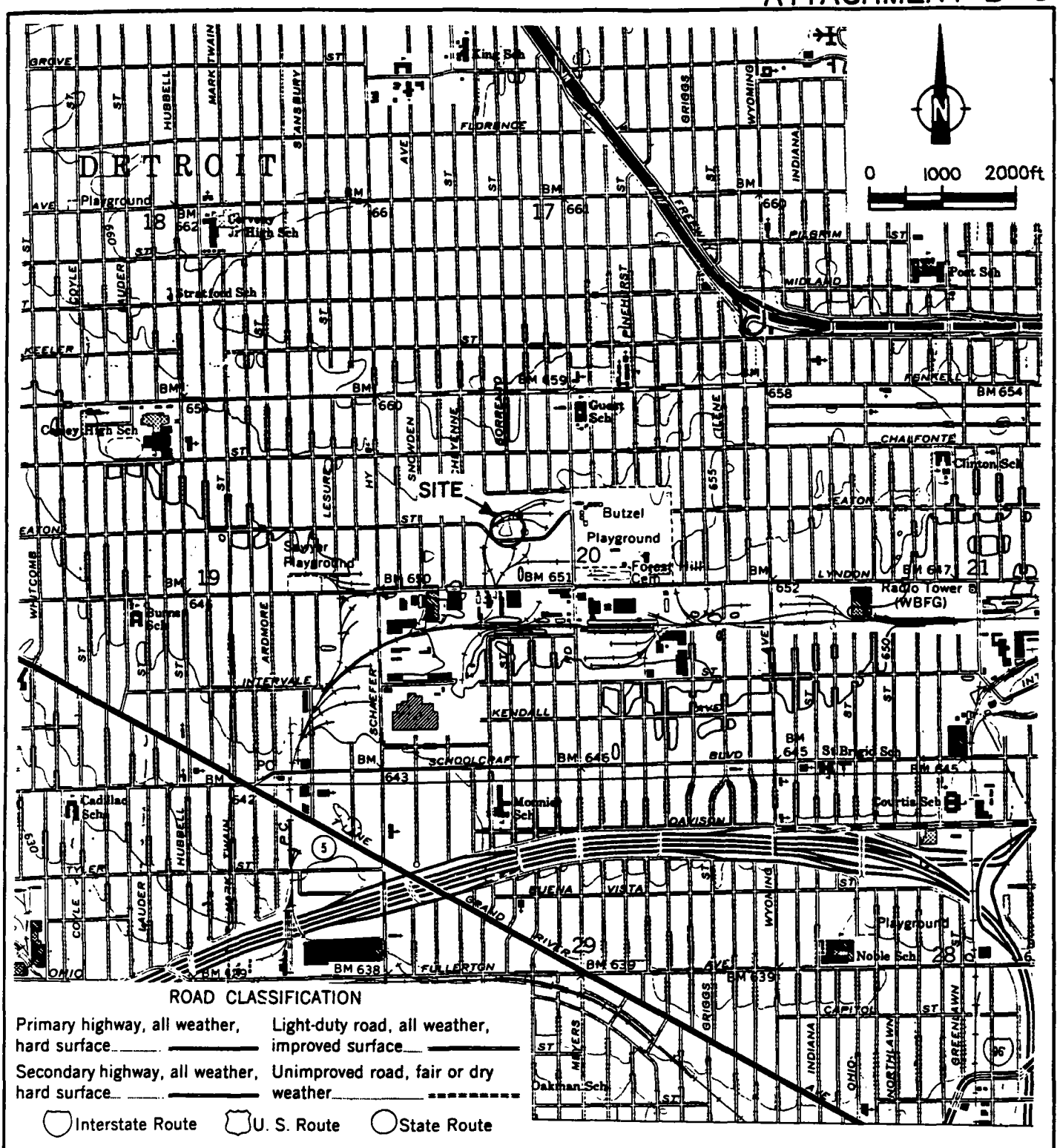
2471-28/07/88-1-D-1

ATTACHMENT B-2

FACILITY TOPOGRAPHIC MAP

ATTACHMENT B-3

SURROUNDING AREA TOPOGRAPHIC MAP



ROYAL OAK, MICH.
N4222.5—W8307.5/7.5

1968
PHOTOREVISED 1981
DMA 4368 I NW—SERIES V862

SURROUNDING AREA TOPOGRAPHIC MAP

Detrex Corporation
Eaton Avenue Facility, Detroit

CRA

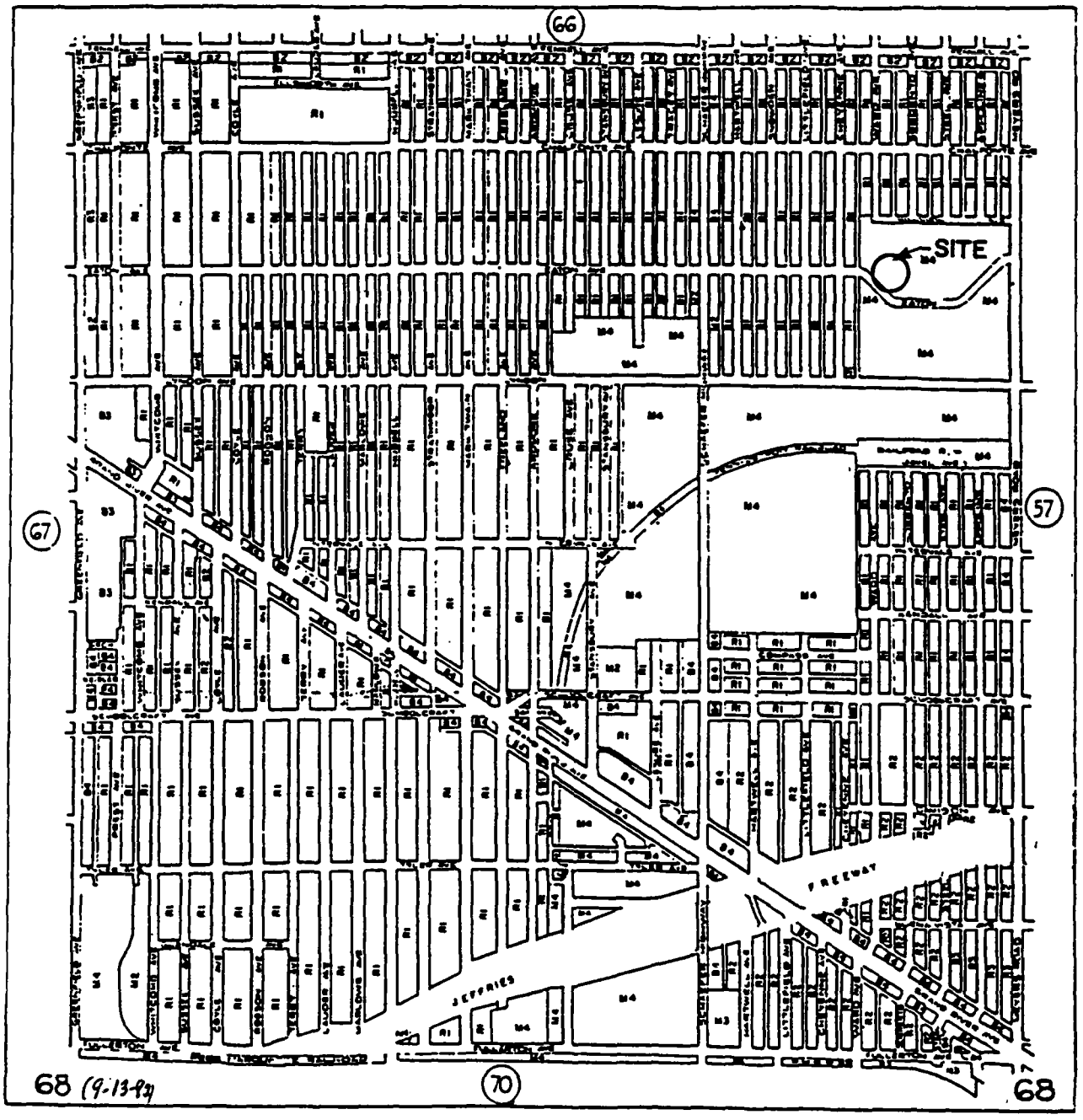
2471-28/07/88-1-D-1

ATTACHMENT B-4

ZONING PLAN

DETROIT ZONING ORDINANCE

N. T. S.



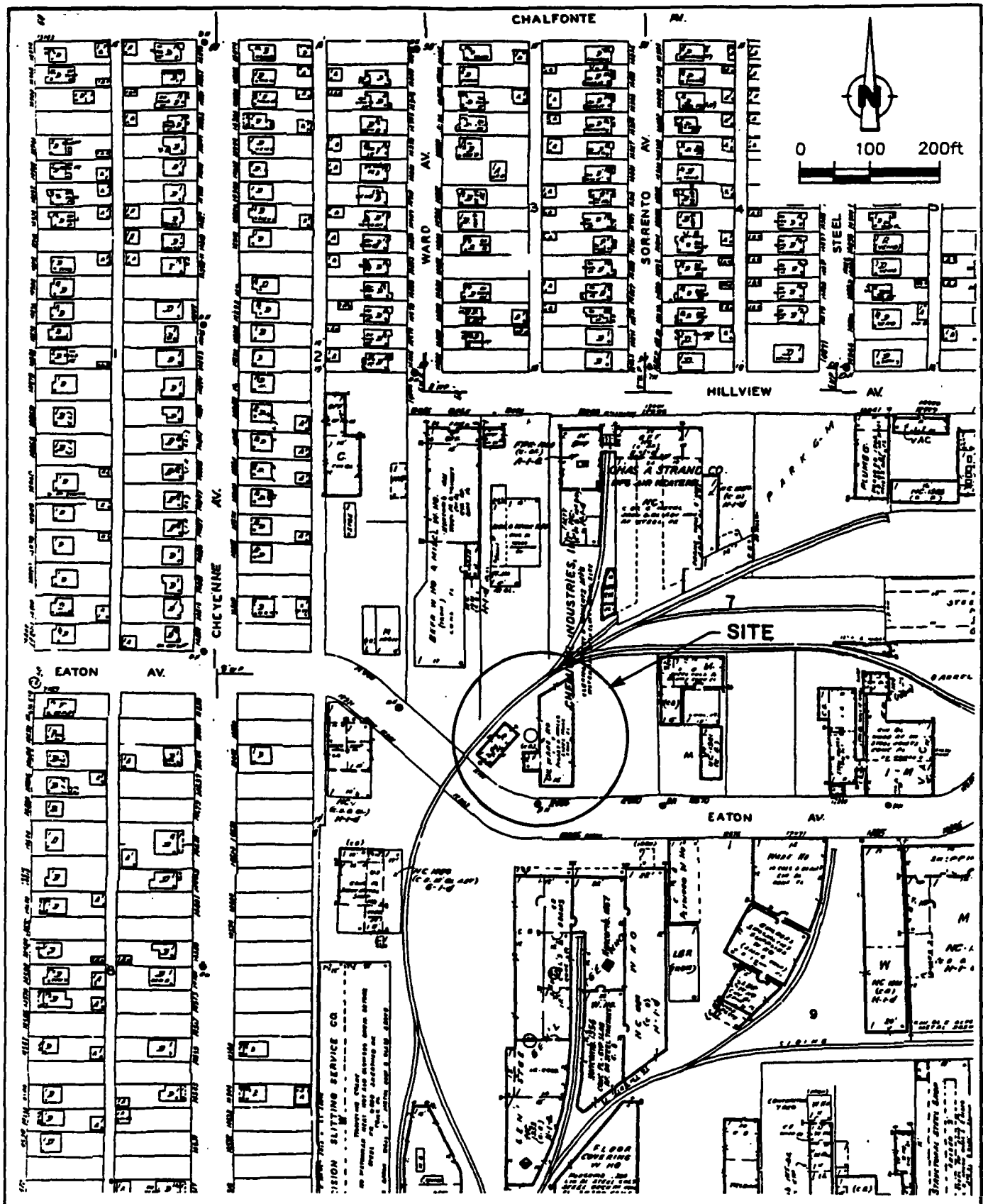
ZONING MAP
Detrex Corporation
Eaton Avenue Facility, Detroit

CRA

2471-28/07/88-1-D-1

ATTACHMENT B-5

LAND USE PLAN



SURROUNDING LAND USE
Detrex Corporation
Eaton Avenue Facility, Detroit

CRA

2471-28/07/88-1-D-1

ATTACHMENT B-6

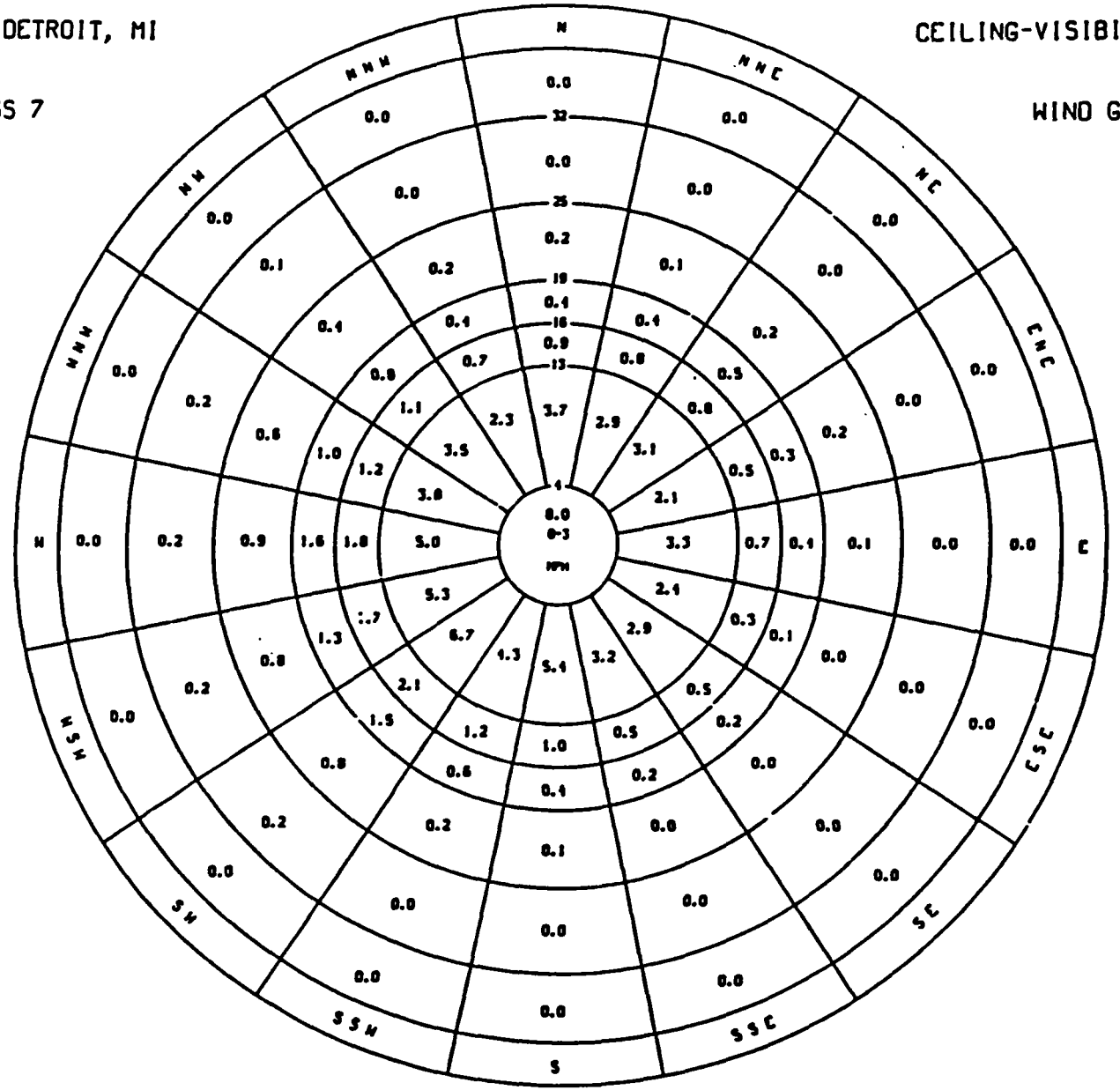
WIND ROSE

DTW DETROIT, MI

CLASS 7

CEILING-VISIBILITY

WIND GRAPH



WIND ROSE

Detrex Corporation

Eaton Avenue Facility, Detroit

CRA

2471-20/06/91-1-0

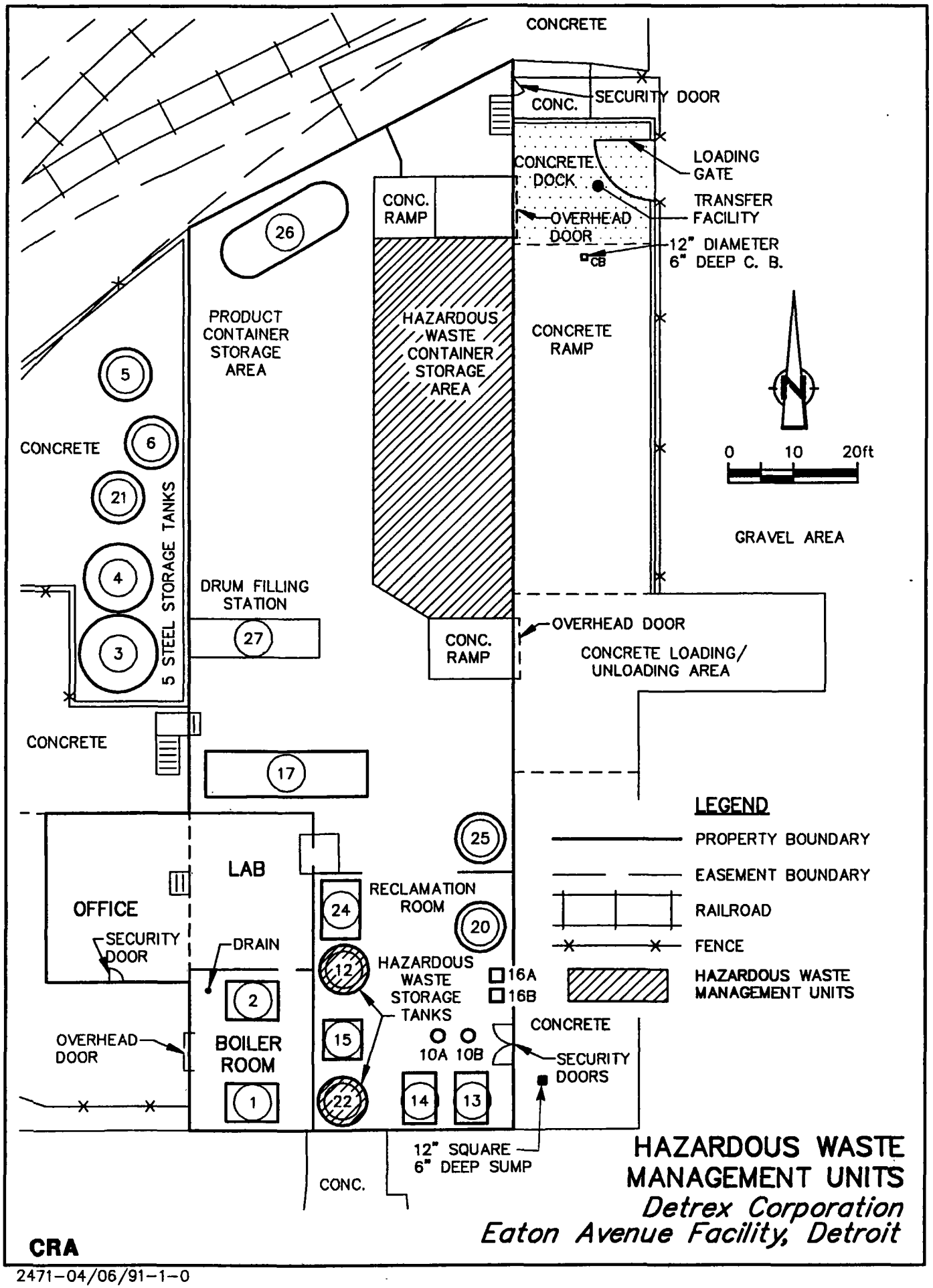
ATTACHMENT B-6

ATTACHMENT B-7

LEGAL SURVEY

ATTACHMENT B-9

HAZARDOUS WASTE MANAGEMENT UNITS



HAZARDOUS WASTE MANAGEMENT UNITS

Detrex Corporation

Eaton Avenue Facility, Detroit

CRA

2471-04/06/91-1-0

ATTACHMENT B-10

FEDERAL INSURANCE ADMINISTRATION

(FIA) FLOOD MAP

SECTION C

WASTE CHARACTERISTICS

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- C-1b Waste Screening C-6
- C-1c Waste Compatibility C-7

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ATTACHMENT C-4	SAMPLE LABORATORY REPORTS
ATTACHMENT C-5	QUALITY ASSURANCE PROJECT PLAN
ATTACHMENT C-6	STANDARD OPERATING PROCEDURE FOR WASTE SAMPLING

SECTION C

WASTE CHARACTERISTICS

This section provides a description of the chemical and physical nature of the hazardous wastes managed in the container and tank storage areas at the Detrex Corporation facility located in Detroit, Michigan.

The Eaton Avenue facility typically receives halogenated solvent wastes for reclamation (recycling) in both drums and bulk shipments from such industries as metal working industries degreasing operations, rubber molding recovery operations, electronic circuit board defluxing operations, and paint spraying equipment cleaning operations. The facility also sells virgin solvents, consequently, personnel are familiar with the wastes that are received at the facility.

The hazardous waste management operations include container storage and tank storage. Wastes are received in container or bulk shipments. The wastes are processed through the recovery (recycling) system which primarily consists of distillation followed by drying. The recovered solvent product is drummed or stored in product tanks for re-sale and the still bottoms remaining after distillation are transferred to a 5,000 gallon generator accumulation tank. The still bottoms are accumulated for less than 90 days prior to shipment off site to a permitted facility.

The information is provided pursuant to Michigan Act 64 Rule 299.9504(1)(c) which incorporates 40 CFR 270.14(b)(2) and (3) by reference. The applicable section(s) of the Federal Regulations (40 CFR) is referenced as appropriate.

C-1 OVERVIEW OF WASTE ANALYSIS PLAN

Detrex Corporation has developed a Waste Analysis Plan to ensure the Eaton Avenue facility accepts only wastes for which the facility is permitted to receive. The Waste Analysis Plan has been prepared recognizing the nature of the wastes accepted by Detrex and based on extensive Corporate experience in the handling and recovery (i.e. recycling) of solvent wastes. A copy of the Waste Analysis Plan is maintained at the facility at all times. The Waste Analysis Plan consists of two major types of characterization.

C-1a Waste Characterization

Prior to accepting wastes from a potential new client, Detrex Corporation requires the potential client to complete and certify a "Generator's Waste Material Profile Sheet". A copy of this profile sheet is included as Attachment C-1. This profile sheet requires the waste generator to complete a chemical analysis of the waste. Detrex then collects a representative sample of the potential clients' waste stream and conducts an analytical verification/confirmation program as a secondary check on the accuracy of the Generator's documented profile sheet. Detrex Corporation reviews the profile sheet and confirmation analysis report and conducts a thorough waste generation process review at the potential clients' facility. This process review pays particular attention to the clients solvent use and solvent handling procedures that generate the waste. Based on Detrex's extensive experience in the industry, this process review, in addition to the waste profile sheet information, is sufficient to enable Detrex to make a determination that the potential clients' waste stream is acceptable for storage and processing at the solvent recovery operation.

As an ongoing process, Detrex is presently requesting Generator Waste Material Profile Sheets from all existing clients. A copy of the "call-in" letter is provided in Attachment C-1. This program will be conducted annually for all clients as a continual check on the wastes being received at the Eaton Avenue facility. At any time that a generator notifies Detrex of a change in the generation process of a waste stream, or at any time that Detrex notices a potential change in waste stream characterization based on regular waste sampling and analysis, a re-characterization program consistent with the program described above will be completed prior to accepting any further wastes from that generator. A record of the results of any annual or other necessary re-characterization review will be maintained on-site in the generator's waste profile files.

C-1b Waste Screening

The second type of waste characterization includes waste screening that is conducted on every shipment of waste that is received at the facility.

Every container or bulk shipment of waste which is received at the facility is sampled. Visual observations of the sample identifing any out of the ordinary observations are recorded. For drum sampling, a composite sample of up to a maximum of 10 drums of a particular waste type per individual generator is then collected for analytical testing prior to signing the manifest. Analytical testing may be done in the on Site laboratory or at another laboratory. The testing procedures described in the following sections are followed by either laboratory. A laboratory Quality Assurance Project Plan (QAPP) has also been developed to ensure the reliability and accuracy of analytical results.

Analytical testing consists of specific gravity, ignitability, and gas chromatography. It is to be noted that ignitability testing is conducted on a discrete sample collected from every container or bulk shipment of waste received at the facility. The analytical results are recorded on an analytical report. The lab report, in conjunction with sample observation and a review of the specific generator's past analysis and waste profile documentation enable Detrex to determine that a waste is acceptable.

Based on the above, approval is given to sign the manifest and accept the wastes into the container or tank storage areas. In the event a waste stream is unacceptable, the manifest is not signed and the original waste generator contacted to determine appropriate handling of the rejected waste. The waste would be returned by Detrex to the original generator or the manifest and land disposal restriction certificate corrected as appropriate and the waste transported to properly permitted facility on behalf of the generator.

A complete record of all sampling and analytical work is placed in the operating files at the facility.

C-1c Waste Compatibility

Another important aspect in the management of hazardous wastes is waste compatibility.

A detailed waste compatibility review was undertaken by Detrex Corporation in accordance with U.S. EPA guidance documents. A copy of this review is provided in Attachment C-4. The compatibility review included the major waste constituents, general blending and stabilizer compounds as well as other potential contaminants that may reasonably be expected to be in the wastes received by Detrex. The review concluded that waste compatibility of wastes that are accepted after screening analyses is not a concern.

It is important to recognize that Detrex has handled these types of solvent wastes for over 25 years without any incidences relating to waste incompatibility. The waste analysis program followed by Detrex does not specifically address waste compatibility, however, during sampling, waste compatibility is evaluated. During the preparation of any composite sample, observations of visual characteristics are noted. Such visual characteristics would include any visual evidence of waste incompatibility (i.e. reaction) noticed in the composite sample as consecutive discrete samples are added to the composite sample. This is similar to procedures that would be followed during a formal on-site waste compatibility testing program. Any discrete sample added to the composite sample that indicates potential evidence of waste incompatibility would be grounds for rejection of the specific drum. Further discrete sampling and analysis of the particular drum or other container noted to be potentially incompatible with the remainder may be conducted to determine the exact source of the incompatibility. The generator would be notified and the generation process re-evaluated to determine future waste acceptance.

In conclusion, any container or bulk shipment which is approved for acceptance at the facility based on the waste analysis procedures described in this document will not create a potential waste incompatibility concern during processing.

C-2 CHEMICAL AND PHYSICAL ANALYSES
[40 CFR §270.14(b)(2), §264.13(a)]

Provided in Table C-1 is a list of all hazardous wastes which may be received and stored in the container or tank storage areas. In addition to the list of wastes which may be stored at the facility, Table C-1 also provides, for each waste, its EPA hazardous waste identification number, the hazardous constituent/characteristic which designates the waste as hazardous, the EPA process code, and the waste's physical state.

Provided as Attachment C-2 to this application are representative Material Safety Data Sheets for each of the solvent hazardous wastes listed in Table C-1. These sheets describe in detail the chemical and physical properties of each waste managed at the facility to allow their safe handling in accordance with 40 CFR Part 264.

The following Sections document the specific analytical procedures followed by Detrex Corporation to ensure only F001 and F002 wastes with the inclusion of D-series compounds under the TCLP rule, are accepted at the facility.

TABLE C-1

LIST OF HAZARDOUS WASTES

Hazardous Waste	*EPA Hazardous Waste Number* (1)	*Hazardous Constituent/Characteristic*	*EPA Process Code*	*Physical State*
1,1,1 Trichloroethane	F001	Toxic	S01/S02	Liquid
Trichloroethylene	F001	Toxic	S01/S02	Liquid
Perchloroethylene	F001	Toxic	S01/S02	Liquid
Methylene chloride	F001	Toxic	S01/S02	Liquid
Trichlorotrifluoroethane (Freon)	F001	Toxic	S01/S02	Liquid
1,1,1 Trichloroethane	F002	Toxic	S01/S02	Semi-Liquid*
Trichloroethylene	F002	Toxic	S01/S02	Semi-Liquid*
Perchloroethylene	F002	Toxic	S01/S02	Semi-Liquid*
Methylene chloride	F002	Toxic	S01/S02	Semi-Liquid*
Trichlorotrifluoroethane (Freon)	F002	Toxic	S01/S02	Semi-Liquid*

(1) Detrex recognizes, based on process knowledge, that these wastes may also be classified as one of the following D-series compounds based on Toxicity Characteristic Leaching Procedure: D004(Arsenic), D005(Barium), D006(Cadmium), D007 (Chromium), D008 (Lead), D009 (Mercury), D010 (Selenium), D011 (Silver), D018 (Benzene), D019 (Carbon Tetrachloride), D021 (Chlorobenzene), D022 (Chloroform), D027 (1,4-Dichlorobenzene), D028 (1,2-Dichloroethane), D029 (1,1-Dichloroethylene), D030 (2,4-Dinitrotoluene), D034 (Hexachloroethane), D035 (Methyl Ethyl Ketone), D036 (Nitrobenzene), D038 (Pyridine), D039 (Tetrachloroethylene), D040 (Trichloroethylene) and D043 (Vinyl Chloride).

* Note: Represents a waste stream that has been partially distilled at the customer's degreasing operation facility.

C-3 WASTE ANALYSIS PLAN [40 CFR §270.14(b)(3), §264.13(b),(c)]

C-3a Parameters and Rationale [40 CFR §264.13(b)(1)]

C-3a (1) Initial Waste Characterization

Profile Information

A Generator's Waste Material Profile Sheet (see Attachment C-1) is required from each potential customer for each type of waste. The information on this form includes the process description, shipping mode, physical properties, chemical properties and certification. Further waste characterization/confirmation is completed by Detrex after receipt of a complete and certified profile sheet.

Analytical Information

As part of the information requested on the profile sheet, the waste generator is required to conduct and complete a chemical analysis of the waste. Detrex Corporation is responsible for collecting a representative sample of the prospective waste stream with analysis then being conducted on the raw waste sample for the following parameters:

Comprehensive Testing

- Ignitability (D001) - EPA SW-846 Method 1010
- Corrosivity (D002) - EPA SW-846 Method 1040
- Reactivity (D003) - EPA SW-846 Section 7
- TCLP Analysis - EPA SW-846 Method 1311

This testing requirement applies only to those generators who have not profiled their material as listed waste (F001, F002) and have not provided characterization of D-series compounds based on process knowledge (i.e. TCLP testing is required for generators who do not classify the TCLP compounds based on analysis or process knowledge). For generators who are profiling listed (process defined) wastes and declaring D-series compounds based in process knowledge, further analysis for the D-series characteristics parameters is not required.

It should be noted that in the event testing is required for copper and zinc, the extraction procedure (EP) toxicity testing method will be utilized until such a time that Michigan receives authority for the TCLP provisions.

Due to the nature of the solvents and processes involved in waste streams submitted to Detrex for recycling, the remaining D-series constituents are not applicable to cleaning/degreasing systems:

D012	Endrin	D025	P-Cresol
D013	Lindane	D026	Cresol
D014	Methoxychlor	D031	Heptachlor
D015	Toxaphene	D032	Hexachloroebenzene
D016	2, 4-D	D033	Hexachloro-1,3-Butadiene
D017	2,4,5-TP Silvex	D037	Pentachlorophenol
D020	Chlordane	D041	2,4,5-Trichlorophenol
D023	O-Crescol	D042	2,4,6-Trichlorophenol
D024	M-Cresol		

These compounds are therefore not required to be formally analyzed via TCLP. Rather Detrex will obtain written certification documentation from the generator verifying that these compounds are not present in the waste stream, based upon the generator's knowledge of the materials and generating processes involved. In the event such certification is not provided, analysis will be conducted by Detrex.

Certain D-series parameters also appear in the 40 CFR 261 listings (process-defined) for F003, F004, and F005 wastes, i.e.

Benzene:	D019 and F003
Methyl ethyl ketone:	D035 and F005
Nitrobenzene:	D036 and F004

Therefore, in such cases where a TCLP analysis or generators process knowledge reveals the presence of these compounds, the generator must provide additional verification of the source (usage) of these compounds, so as to determine whether the waste is to be classified as F003, F004, or F005 on the basis of the generating process.

Fingerprint Testing

In addition, a laboratory-scale estimate of potential solvent recovery and purity is performed via rotary vacuum distillation and specific gravity testing. The overhead distillate (recovered solvent) is subsequently analyzed by Gas Chromatography.

Process Review Verification

As the final step in the pre-acceptance qualification procedure, Detrex Corporation conducts an on-Site waste generation process review at the potential client's facility. In examining the generator's solvent usage and handling procedures, Detrex confirms proper classification of the waste with reference to listing description/definitions set forth in 40 CFR part 261. Only F001 and F002 listed waste types are acceptable for storage and processing at the Detrex solvent recovery operations.

This process review, in addition to the profile and analytical information as presented above enable Detrex to make a thorough and accurate determination of the characteristic and/or listed classification of each submitted waste stream, along with subsequent final acceptability of each stream in accordance with the facility permit. Documentation of waste characterization is retained on file, on a per-waste stream basis, at the facility.

If an incoming waste is properly characterized as a F001 or F002 waste stream, it should be acceptable to the process stream. The solvent content of the waste, as determined by rotary vacuum distillation and the specific gravity method, provides an estimate of the recoverable solvent yield from the waste stream. Thus, as long as a waste is properly characterized as a F001 or F002 waste, with the inclusion of D-series compounds under the TCLP rule the waste is physically acceptable to the process unit. There are no particular tolerance limits for acceptable versus unacceptable wastes.

C-3a (2) Waste Screening

Waste screening analysis is conducted on all wastes received at the facility to confirm the wastes acceptance prior to signing the manifest. The screening analysis is summarized on Table C-2 with parameter and rational for selection.

C-3b Test Methods [40 CFR §264.13(b)(2), §264.13(c)(1)]

Detrex is able to conduct all waste screening analysis at the Eaton Avenue facility. More detailed waste characterization is completed at an off-site laboratory facility following the Detrex Waste Analysis Plan. An off-site facility may also conduct screening analysis as conditions require (equipment maintenance). All containers are stored in a maintained segregated area within the hazardous waste container storage area until analysis is complete and the manifest is signed.

Detrex will conduct an ignitability test (ASTM Method D4982-89) on a discrete sample collected from each containers or bulk shipment of waste received at the facility. In the event a positive result is

TABLE C-2

PARAMETERS AND RATIONALE FOR THEIR SELECTION

Classification	*Parameter*	*Rationale*
F001 Hazardous Waste	1,1,1 Trichloroethane Trichloroethylene Perchloroethylene Trichlorotrifluoroethane Methylene Chloride	Listed toxic waste (F001)
F002 Hazardous Waste	1,1,1 Trichloroethane Trichloroethylene Perchloroethylene Trichlorotrifluoroethane Methylene Chloride	Listed toxic waste (F002)
Stabilizer Compounds	2-Propanol Nitromethane Butylene Oxide sec - Butyl Alcohol Dioxolane Cyclohexeneoxide n-propanol Toluene 1,4-Dioxane	Processing Knowledge (to determine amount/type of additional stabilizer required after recycling)
Other Compounds	Methanol Ethanol Acetone Methyl Ethyl Ketone Methylisobutyl Ketone t-Butyl Alcohol n-Butyl Alcohol Toluene Xylenes	Processing Knowledge [Expected only in very small quantities (ie. much less than 10 percent)]
	Ignitability	To ensure no flammable wastes are accepted
	Solvent Content (%)	Processing Knowledge
	Non-Volatile Residue (%)	Processing Knowledge

observed, the specific container(s) or bulk waste shipment will be rejected and immediately returned to the original generator by Detrex or the manifest and land disposal restriction certificate corrected as appropriate and the waste transported off Site to a permitted facility on behalf of the original generator. The waste would further tested utilizing ASTM D93 (Test Method for Flash Point by Pensky-Martens Closed Test) to confirm a flash point below 140°F in the event the manifest is to be changed to ensure proper classification

Provided as Attachment C-3 are copies of the Standard Operating Procedures for all testing methods conducted by Detrex. Included is the specific gravity test; a copy of ASTM Method D4982-89 (ignitability test); the solvent extraction method; and method for determination of solvents in spent solvents by gas chromatography. The solvent extraction and gas chromatography methods were developed by Detrex Corporation. They are modifications to U.S. EPA approved methodologies (i.e. Methods 8010 and 8015, SW-846). Standard methodologies are not appropriate due to the percentage levels of organics present in the waste streams. Standard methodologies were developed for the purpose of evaluating concentrations at very low detection levels for drinking water purposes.

As previously discussed in Section C-1c, Detrex has thoroughly reviewed waste compatibility in accordance with the document "A Method of Determining the Compatibility of Hazardous Waste, EPA-600/2-80-076, April 1980". Based upon this review and general knowledge of these wastes managed by Detrex for over 25 years, Detrex believes potential waste incompatibility is not a concern and thus proposes no further ongoing waste compatibility testing unless a new parameter for evaluation becomes evident. In the event of any parameter changes, Detrex will readdress waste compatibility.

Detrex also conducts a visual inspection of the hazardous waste during screening of the waste. Observations are made during sampling pertaining to abnormal solid content, color, or if it is an aqueous solution rather than an oil/solvent mixture.

Attachment C-4 presents a copy of the analytical report generated during waste screening. All appropriate laboratory results are documented on the laboratory report. In order to ensure the reliability and accuracy of the analytical results, Detrex Corporation has developed a Quality Assurance Project Plan (QAPP). A copy of this document is provided as Attachment C-5.

This document describes all laboratory procedures and related quality control/quality assurance procedures followed by Detrex Corporation laboratory personnel to ensure the reliability of analytical results.

C-3c Sampling Method [40 CFR §264.13(b)(3), §264.13(c)(2)]

The collection of concentrated waste samples is conducted by facility personnel. The samples are collected by glass thief. Sampling procedures are described in Attachment C-6.

C-3d Frequency of Analysis [264.13(b)(4]

Waste screening as described in the waste analysis plan is conducted on each shipment of wastes received from a customer.

The waste is sampled as per the sampling method presented in Section C-3c. A preliminary assessment of the waste sample will be made by running a rotary vacuum distillation and specific gravity test to determine the approximate solvent content and an ignitability test to ensure it is not potentially flammable. The sample is then submitted for organic analysis. Analytical procedures were described in Section C-3b and are further described in the QAPP.

Regardless of whether or not Detrex accepts the waste for processing, all of the foregoing knowledge, including laboratory results, is kept in the customer's file for future reference and comparison with future shipments, if they become a regular customer.

C-3e Additional Requirements for Wastes Generated Off Site [40 CFR § 264.13(b)(5)]

Each waste stream will be re-characterized annually, at a minimum, or when Detrex is notified or has reason to believe that the process or operation generating the hazardous waste has changed; or when the results of waste shipment inspections indicate that the hazardous waste received at the facility does not match the waste designated on the accompanying manifest. Detrex has implemented procedures requiring generators to notify Detrex when operation procedures change.

ATTACHMENTS

ATTACHMENT C-1

GENERATORS WASTE MATERIAL PROFILE SHEET

For Office Use Only

DETREX CORPORATION
Solvents Division
12886 EATON AVE. DETROIT, MI 48227
(313) 491-4550 FAX:(313) 491-8044

GENERATOR'S WASTE MATERIAL PROFILE SHEET

Code: _____

Date: _____

Sales: _____

IMPORTANT: BEFORE WE CAN ACCEPT HAZARDOUS WASTE FOR PROCESSING OR DISPOSAL, WE MUST ANALYZE A CROSS SECTIONAL SAMPLE UNIQUE TO EACH WASTE STREAM AND TRULY REPRESENTING ALL PHYSICAL AND CHEMICAL PROPERTIES. IT IS EXTREMELY IMPORTANT, THEREFORE, THAT PROPER SAMPLING TECHNIQUE BE USED AND THAT THE SAMPLE BE DRAWN BY A GENERATOR'S EMPLOYEE FAMILIAR WITH BOTH THE WASTE STREAM AND SAMPLING TECHNIQUE. IN CASES WHERE THE SAMPLING IS DONE BY PERSONNEL OTHER THAN GENERATOR EMPLOYEES, A QUALIFIED GENERATOR EMPLOYEE SHALL WITNESS AND DIRECT THE SAMPLING PROCEDURE, AND SIGN THIS FORM IN THE APPROPRIATE PLACE. CHEMICAL ANALYSIS COMPLETED BY A REPUTABLE INDEPENDENT LABORATORY WILL BE ACCEPTED PROVIDING THE ANALYSIS IS IN SUFFICIENT DETAIL AND THE GENERATOR ASSURES US THAT PROPER SAMPLING TECHNIQUE WAS USED. PLEASE TYPE OR COMPLETE THIS FORM IN INK FOR EACH SAMPLE AND RETURN IT TO US AT THE ADDRESS CHECKED OFF ON THE REVERSE SIDE OF THIS FORM. PHOTO COPY FOR YOUR RECORDS.

A. GENERAL INFORMATION

Generator Name _____ Generator USEPA ID: _____

Facility Address: _____ Generator State ID: _____

_____ Technical Contact: _____

_____ Title: _____

Phone: _____ FAX: _____

B. MAIL INVOICES TO

Company Name: _____ Business Contact: _____

Address: _____ Title: _____

_____ _____

Phone: _____ FAX: _____

C. WASTE STREAM DESCRIPTION

Generators Common Name or Description of Waste Product _____

Process Description _____

OTE: Information	☐ Under 50 PPM	☐ Between 50 - 500 PPM	☐ Over 500 PPM	☐ Solid	☐ Liquid	☐ Not Present
Pesticide Information	☐ Under 50 PPM	☐ Between 50 - 500 PPM	☐ Over 500 PPM	☐ Solid	☐ Liquid	☐ Not Present
PCP Information	☐ Under 50 PPM	☐ Between 50 - 500 PPM	☐ Over 500 PPM	☐ Solid	☐ Liquid	☐ Not Present
Dioxin Information	☐ Under 50 PPM	☐ Between 50 - 500 PPM	☐ Over 500 PPM	☐ Solid	☐ Liquid	☐ Not Present

D. PROJECTED VOLUME:

_____ Gals Per: ☐ One Time, ☐ Week, ☐ Month, ☐ Quarter, ☐ Year

Quantity On Hand _____ Generator Preference: ☐ Recycle and Return ☐ Disposal

E. SHIPPING AND HANDLING REQUIREMENTS

Mode of Packaging: ☐ Drum ☐ Bulk ☐ Liquid ☐ Solid ☐ Other _____

Size of Container(s) _____

Transfer Equipment Requirements (Dusty, Odiferous, Toxic Volitile Fumes, Etc.): _____

Special Requirements: _____

F. HAZARDOUS PROPERTIES

USEPA Hazardous Waste? ☐ YES ☐ NO USEPA Hazardous Code(s) _____

State Hazardous Waste? ☐ YES ☐ NO State Code(s) _____

Reactivity: ☐ None ☐ Pyrophoric ☐ Shock Sensitive ☐ Explosive ☐ Water Reactive ☐ Other _____

Other Hazardous Characteristics:

☐ None ☐ Radioactive ☐ Etiological ☐ Pesticide Manufacturing Waste ☐ Other _____

Is this waste product a hazardous material as defined by the United States Department of Transportation pursuant to the hazardous material transportation act?* ☐ Yes ☐ No

If yes, please specify the following:

Proper D.O.T. Shipping Name: _____

D.O.T. Hazard Class: _____

D.O.T. Identification Number(s): _____

D.O.T. Shipping Container(s): _____

*(SEE CFR 49 PART 172.101 FOR HAZARDOUS MATERIALS LIST AND CHARACTERISTICS)

Color: _____

Odor: ☐ None ☐ Mild ☐ Strong

Describe _____

Physical State at 70° F:

☐ Solid ☐ Liquid ☐ Powder

Phases or Layers:

☐ Homogeneous ☐ Bilayered ☐ Multi-Layered

Viscosity:

☐ Low ☐ Medium ☐ High

Solids: ☐ By Weight ☐ By Volume

Total _____%, Dissolved _____%, Suspended _____%

Free Liquids: ☐ Yes ☐ No Volume _____%

Weight: _____

☐ Lbs./Gallons (Liquid) ☐ Lbs./Cubic Foot (Solids)

% Solvent Recovery _____%

Still Bottom Type: ☐ Resinous ☐ Oily

Specific Gravity: ☐ 0.65—0.80 ☐ 0.81—0.95 ☐ 0.96—1.05 ☐ 1.06—1.20 ☐ 1.21—1.40 ☐ 1.41—1.70 ☐ 1.70+ ☐ Exact _____

Flash Point: ☐ 70° F. ☐ 70°F—100°F ☐ 101°F—139°F ☐ 140°F—200°F ☐ 200°F ☐ No Flash ☐ Closed Cup ☐ Open Cup ☐ Exact _____

Melt Point: _____ °F

BTU Value: _____ per pound _____ per gallon

pH (Indicate Range)

☐ 2.0 ☐ 2.1—4.0 ☐ 4.1—6.9 ☐ 7.0 ☐ 7.1—10 ☐ 10.1—12.5 ☐ 12.5 ☐ N/A ☐ Exact _____

H. CHEMICAL COMPOSITION (Totals Must Add to 100%)

Water Content _____%

_____ _____%

_____ _____%

_____ _____%

_____ _____%

_____ _____%

_____ _____%

_____ _____%

_____ _____%

Metals ☐ Total (PPM) ☐ EPA Extraction Procedure (mg/L)

Arsenic (As) _____ Selenium (Se) _____

Barium (Ba) _____ Silver (Ag) _____

Cadmuim (Cd) _____ Copper (Cu) _____

Chromium (Cr) _____ Nickel (Ni) _____

Mercury (Hg) _____ Zinc (Zn) _____

Lead (Pb) _____ Chromium-Hex (Cr + 6) _____

Other Components - Total (PPM)

Nitrogens _____

Cyanides _____ Halogens _____

Sulphur _____ Phenolics _____

I. SAMPLING INFORMATION:

Date Sampled: _____ Time of Day _____ Temperature _____ °F

Type of Container: _____ Size: _____

Type of Sampling Device: _____

Were the Container and Sampling Device Clean? ☐ Yes ☐ No Sample Taken From: ☐ Drum ☐ Tank ☐ Other _____

Method of Sampling: _____ IF&R Label Affixed To Sample? ☐ Yes ☐ No

Personnel: Sampler - (Please Print) _____

Waste Product Stored: ☐ Inside ☐ Heated ☐ Not Heated ☐ Outside ☐ Outside But Under Roof

J. SPECIAL HANDLING INFORMATION _____

_____

_____

_____ ☐ Additional Page(s) Attached

K. CERTIFICATION:

I THE UNDERSIGNED, AN EMPLOYEE OF THE GENERATOR AND HAVING PROPER AUTHORITY GRANTED BY THE GENERATOR, HEREBY CERTIFY THAT THE SAMPLE DEFINED HEREIN IS A TRUE CROSS SECTIONAL SAMPLE REPRESENTING ALL PHYSICAL AND CHEMICAL PROPERTIES OF THE WASTE STREAM AS DEFINED IN PARAGRAPH B HEREIN, AND THAT I HAVE EXAMINED AND AM FAMILIAR WITH THE INFORMATION SUBMITTED IN THIS AND ALL ATTACHED DOCUMENTS, AND THAT TO THE BEST OF MY KNOWLEDGE IT IS TRUE AND CORRECT, AND THAT ALL KNOWN AND SUSPECTED HAZARDS HAVE BEEN DISCLOSED.

_____ GENERATOR PERSONNEL (SIGNATURE) _____ (TITLE) _____ (DATE)

_____ DISTRIBUTOR SALESMAN (PLEASE PRINT) _____ (DATE)

L. WASTE SAMPLE QUALIFICATION FEE: $_____

Customer P.O. #_____ _____ (SIGNED)

SEND SAMPLE AND WASTE PROFILE TO: DETREX CORPORATION 12886 Eaton Ave., Detroit, MI 48227
Solvents Division

GSF 8905.4 d (2/2)

DETREX CORPORATION
Solvents Division
12886 Eaton Ave.
DETROIT, MI 48227

FAX:(313) 491-8044

TELEPHONE:
(313) 491-4550

February 6, 1991

During the past three years, the United States Environmental Protection Agency (EPA) has promulgated a series of new regulations pertaining to the treatment, storage, disposal, classification and testing of hazardous waste. Detrex Corporation, Solvents Division has maintained all current required regulatory documentation throughout this period, for the purpose of protecting its customers from any potential liability risk, while still continuing to provide comprehensive environmental services.

In an effort to maintain these standards of service it is necessary to initiate the updating of all waste stream information records. We are asking all of our customers to complete the enclosed "Generator's Waste Material Profile Sheet", making particular note of the following areas:

1. Technical Contact (Sec. A)

   This must be the person responsible for providing all of the correct waste stream and shipment information.

2. Complete Process Description (Sec. C)

   Please detail the waste generation process, including any "special" aspects of your operation that affect the waste composition.

3. USEPA Hazardous Codes (Sec. F)

   Note that in addition to your primary F 001 and/or F 002 listing, new D 040 (trichloroethylene) and D 039 (perchloroethylene) codes also apply as of September 25, 1990.

   Example land - ban notification forms are included for reference.

4. USDOT Shipping Name (Sec. F)

   Example manifests are also enclosed for your waste type(s), disregard the descriptions which do not apply to your waste stream. Use these for shipping reference as well as for correct filling of the Waste Material Profile Sheet section.

5. Technical Contact Signature (Certification: Sec. K)

   This must be signed for the form to be valid.

Please return the completed Profile Sheet to us, in the enclosed envelope, by February 28, 1991 so that we may expedite the updating of our files. If you have any questions, please contact your Detrex Account Representative or Joe Calderoni, our Quality Control Manager.

Thank you for your cooperation in allowing Detrex to continue to improve its services. We look forward to meeting your product and waste management needs for many more years to come.

Sincerely,

Ronald C. Hritzkowin
Facility Manager

RCH:kh

CC: M.O. O'Connor, Sales Manager
R. Anderson, Account Representative
P. Koehler, Account Representative

ATTACHMENT C-2

MATERIAL SAFETY DATA SHEETS

Material Safety Data Sheet

MSD 8208.22

DETREX CHEMICAL INDUSTRIES, INC.
P.O. BOX 501,
DETROIT, MICHIGAN 48232

Approved by U.S. Dept. of Labor as "Essentially similar" to Form OSHA-20

Date: August, 1982	Edition: First
Chemical Name and Synonyms: Perchloroethylene; tetrachloroethylene CAS No.: 127-18-4	Trade Name and Synonyms: DETREX PERK, Perchlor, Perchloroethylene
Chemical Family: Halogenated Hydrocarbons	Formula: $CCl_2=CCl_2$
DOT Shipping Name: Tetrachloroethylene	DOT Hazard Class: ORM-A-UN1897

SECTION 1 · PHYSICAL DATA

Boiling Point @ 760 mm Hg:	Vapor Density (Air=1):	Specific Gravity (H_2O=1):	pH of Solutions:
250°F 121°C	5.83	(20°/20°C) 1.6	6.8 to 8.4
Freezing/Melting Point:	**Solubility (Weight % in Water):**	**Bulk Density:**	**Volume % Volatile:**
-8.2°F -22.3°C	@25°C 0.015%	13.6 lbs./gal. @ 20°C	Essentially 100
Vapor Pressure:	**Evaporation Rate**	**Heat of Solution:**	**Appearance and Odor**
@ 20°C = 14.2mmHg	(ethyl ether=1): 0.09	Not Applicable	Clear, colorless liquid with ether-like odor.

SECTION 2 · HAZARDOUS INGREDIENTS

	%	Hazard Data
Perchloroethylene (Stabilized)	100	See Sections 4 & 5

SECTION 3 · FIRE AND EXPLOSION HAZARD DATA

Flash Point °F (Method Used)	Flammable Limits in Air (% by Volume)	Extinguishing Media:
None	None LEL: UEL:	For fires involving perchloroethylene, use water, dry chemical or carbon dioxide.

Special Fire Fighting Procedures: Fire fighters should wear pressure-demand self-contained breathing apparatus for possible exposure to hydrogen chloride and possible traces of phosgene.

Unusual Fire and Explosion Hazards: Perchloroethylene involved in fires can decompose to hydrogen chloride and possible traces of phosgene. See Detrex warning letter Form SoL 8208.21 attached.

SECTION 4 · HEALTH HAZARD DATA

Permissible Exposure Limits (TLV): See Section 5

Toxicity Data Ref.(1)	Classification (Poison, Irritant, Etc.)
LCLoInhalation (rat) 4,000 ppm (4 hours)	Inhalation: Moderately Toxic
LD_{50} Dermal	Skin/Eye: Liquid mildly irritating to skin; eye irritant
LD_{50} Ingestion (rabbit) - 5,000 mg/kg	Ingestion: Slightly Toxic
Fish, LC_{50} (Lethal Concentration) 96hr. TLM 100-10 ppm	Aquatic: Toxic

Human Exposure Information/Data: Unconfirmed data[1] exists which indicate that perchloroethylene by ingestion may be more toxic to humans than indicated by the available data. Such unconfirmed data report poisonings at doses as low as 500 mg/kg.

24-HOUR EMERGENCY ASSISTANCE: (313) 358-5800

Section 4(Cont'd)- Permissible Exposure Limits

Current OSHA permissible exposure limits (29CFR 1910.1000) are 100 ppm (8-hour TWA); 100-200 ppm periodic excursions are allowed providing 8-hour TWA is at or below 100 ppm; 200-300 ppm excursions allowed only for maximum of 5 minutes in any 3-hour period; 300 ppm maximum allowable concentration (must not be exceeded).

ECTION 5 · EFFECTS OF OVEREXPOSURE

This section covers effects of overexposure for inhalation, eye/skin contact, ingestion and other types of overexposure information in the order of the most hazardous and the most likely route of overexposure.

Acute: Primarily a central nervous system depressant. Inhalation can cause irritation of the respiratory tract, dizziness, nausea, headache, loss of coordination and equilibrium, unconsciousness and if exposed to high concentrations in confined or poorly ventilated areas, even death.

Liquid splashed in the eye can result in discomfort, pain and irritation. Prolonged or repeated contact with liquid on the skin can cause irritation and dermatitis. The problem may be accentuated by liquid becoming trapped against the skin by contaminated clothing and shoes. Skin absorption can occur.

Chronic: Prolonged exposure above the OSHA permissible exposure limits may result in liver and kidney damage. Perchloroethylene has been extensively studied for cancer both in the U.S. and Europe by government, industry and academia. There is no documented evidence that perchloroethylene causes an increased cancer incidence in humans.

EMERGENCY AND FIRST AID PROCEDURES:

Inhalation: Remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician.

Eye or Skin Contact: Flush eyes and skin with plenty of water (soap and water for skin) for at least 15 minutes, while removing contaminated clothing and shoes. If irritation occurs, consult a physician.

Ingestion: If conscious, drink a quart of water. DO NOT induce vomiting. Take immediately to a hospital or physician. If unconscious, or in convulstions, take immediately to a hospital or physician. DO NOT give anything by mouth to an unconscious person.

Notes to Physician (Including Antidotes): NEVER administer adrenalin following perchloroethylene overexposure. Increased sensitivity of the heart to adrenalin may be caused by over-exposure to perchloroethylene.

SECTION 6 · REACTIVITY DATA

Stability: Stable	**Conditions to Avoid:** Avoid open flames, hot glowing surfaces or electric arc.
Hazardous Polymerization: Will not occur	**Conditions to Avoid:** None

Incompatibility (Materials to Avoid): Avoid contamination with caustic soda, caustic potash or oxidizing materials. Shock sensitive explosives may be formed. Also see Detrex warning letter Form SoL 8208.21 attached.

Hazardous Decomposition Products: Hydrogen chloride and possibly traces of phosgene.

SECTION 7 · SPILL OR LEAK PROCEDURES (See Detrex Forms SoL 8208.14 and SoL 8208.15 attached)

Steps to be Taken if Material is Spilled or Released: Immediately evacuate the area and provide maximum ventilation. Unprotected personnel should move upwind of spill. Only personnel equipped with proper repiratory and skin/eye protection should be permitted in area. Dike area to contain spill. Take precautions as necessary to prevent contamination of ground and surface waters. Recover or absorb spilled material on sawdust or vermiculite and sweep into closed containers for disposal. After all visible traces have been removed, thoroughly wet vacuum the area. DO NOT flush to sewer. If area of spill is porous, remove as much contaminated earth and gravel, etc., as necessary and place in closed containers for disposal. (See Below)

Waste Disposal Method:

Contaminated sawdust, vermiculite or porous surface must be disposed of in a permitted hazardous waste management facility. Recovered liquids may be reprocessed or incinerated or must be treated in a permitted hazardous waste management facility. Care must be taken when using or disposing of chemical materials and/or their containers to prevent environmental contamination. It is your duty to dispose of the chemical materials and/or their containers in accordance with the Clean Air Act, the Clean Water Act, the Resource Conservation and Recovery Act and all relevant state or local laws/regulations regarding disposal.

SECTION 8 · SPECIAL PROTECTION INFORMATION

Respiratory Protection: For emergencies or working in confined areas, wear self-contained breathing apparatus or supplied air respiratory protection (use the "buddy system" and wear a safety harness with a lifeline). In other circumstances involving potential overexposures, use NIOSH/MSHA-approved organic vapor respirator. (Observe limitations specified by manufacturer.) Respiratory protection program must be in accordance with 29CFR 1910.134.

Ventilation (Type): Mechanical (General) - Sufficient to maintain workplace concentration below permissible exposure limits.

Eye Protection: Splashproof goggles	**Gloves:** Polyethylene, neoprene or polyvinyl

Other Protective Equipment: Safety shower and eye-wash fountain in immediate area. Personnel protective clothing and use of equipment must be in accordance with 29CFR 1910.133 and 29CFR 1910.132.

SECTION 9 · SPECIAL PRECAUTIONS

Precautions to be Taken During Handling and Storing:

- Do not use in poorly ventilated or confined spaces.
- Perchloroethylene vapors are heavier than air and will collect in low areas.
- Keep container closed when not in use.
- Do not store in open, unlabeled or mislabeled containers.
- Liquid oxygen or other strong oxidants may form explosive mixtures with perchloroethylene.
- This material or its vapors when in contact with flames, hot glowing surfaces or electric arcs can decompose to form hydrogen chloride gas and traces of phosgene.
- AVOID CONTAMINATION OF WATER SUPPLIES: Handling, storage and use procedures must be carefully monitored to avoid spills or leaks. Any spill or leak has the potential to cause underground water contamination which may, if sufficiently severe, render a drinking water source unfit for human consumption. Contamination that does occur cannot be easily corrected.

Other Precautions:

- AVOID PROLONGED OR REPEATED BREATHING OF VAPORS. High vapor concentrations can cause dizziness, unconsciousness or death. Long-term overexposure may cause liver/kidney injury. USE ONLY WITH ADEQUATE VENTILATION. Ventilation must be sufficient to limit exployee exposure to perchloroethylene below OSHA permissible exposure limit (8-hour TWA - 100 ppm; ceiling 200 ppm; maximum peak 300 ppm, 5 minutes in any 3 hours). Observance of lower limits (outlined in Section 4) is advisable.
- AVOID CONTACT WITH EYES. Will cause irritation and pain.
- AVOID PROLONGED OR REPEATED CONTACT WITH SKIN. May cause irritation or dermatitis.
- DO NOT TAKE INTERNALLY. Swallowing may cause injury or death.
- DO NOT EAT, DRINK OR SMOKE IN WORK AREAS.

References:

1. NIOSH Registry of Toxic Effectos of Chemical Substances, 1978
2. Industrial Hygiene and Toxicology, Volume II, Second Edition, F.A. Patty, 1963
3. Dangerous Properties of Industrial Materials, Fifth Edition, N. I. Sax, 1979
4. Federal Register, 45FR Hazardous Waste Management Systems Part III, Identification and Listing of Hazardous Wastes, Page 33084, May 19, 1980
5. Science Advisory Board, Subcommittee on Airborne Carcinogens, September, 1980

Comments: As this solvent (perchloroethylene) is used to clean and/or degrease a variety of metal and plastic parts, it should always be used in conjunction with properly designed and fully controlled solvent vapor degreasing equipment that is in compliance with the U. S. Environmental Protection Agency, OAQPS Guidelines, "Control of Volatile Organic Emissions from Solvent Metal Cleaning", and/or any other applicable federal, state or local regulatory guidelines.

Perchloroethylene is also used extensively in the commercial drycleaning industry. It should always be used in conjunction with properly designed and fully controlled equipment that is in compliance with all applicable federal, state and local regulatory guidelines.

Material Safety Data Sheet

MSD 8208.20

DETREX CHEMICAL INDUSTRIES, INC.
P.O. BOX 501,
DETROIT, MICHIGAN 48232

Approved by U.S. Dept. of Labor as "Essentially similar" to Form OSHA-20

Date: August, 1982	Edition: First
Chemical Name and Synonyms: 1,1,1-trichloroethane; methylchloroform CAS No. 71-55-6	Trade Name and Synonyms: PERM-ETHANE ® DG
Chemical Family: Halogenated Hydrocarbons	Formula: CH_3CCl_3
DOT Shipping Name: 1,1,1-trichloroethane	DOT Hazard Class: ORM-A I. D. Number: UN 2831

SECTION 1 · PHYSICAL DATA

Boiling Point @ 760 mm Hg:	Vapor Density (Air=1):	Specific Gravity (H_2O=1):	pH of Solutions:
165.4°F	4.54	1.300-1.320 @ 25°/25°C	6.0 to 7.5
Freezing/Melting Point:	**Solubility (Weight % in Water):**	**Bulk Density:**	**Volume % Volatile:**
-49°F -45°C	Negligible	10.80-10.97 lbs/gal @25°C	Essentially 100
Vapor Pressure:	**Evaporation Rate**	**Heat of Solution:**	**Appearance and Odor:**
@25°C = 135mmHg	(ethyl ether = 1):0.35	Not Applicable	Clear, colorless liquid - ether-like odor.

SECTION 2 · HAZARDOUS INGREDIENTS

	%	Hazard Data
1,1,1-trichloroethane (Stabilized)	100	See Sections 4 & 5

SECTION 3 · FIRE AND EXPLOSION HAZARD DATA

Flash Point °F (Method Used)	Flammable Limits in Air (% by Volume)	Extinguishing Media:
None when tested in accordance with DOT requirements.	LEL: 7% UEL: 15% See Below	water, dry chemical or carbon dioxide

Special Fire Fighting Procedures: Fire fighters should wear a NIOSH/MSHA-approved pressure-demand, self-contained breathing apparatus for possible exposure to hydrogen chloride and possibly traces of phosgene. Use water only in degreasers when aluminum reaction occurs.

Unusual Fire and Explosion Hazards: Vapors concentrated in a confined or poorly ventilated area can be ignited upon contact with a spark, flame, or high intensity source of heat. This can occur at concentrations ranging between 7-15% by volume. Decomposition or burning can produce hydrogen chloride or possibly traces of phosgene. Also see Detrex warning letter Form SoL 8208.21 attached.

SECTION 4 · HEALTH HAZARD DATA

Toxicity Data	Classification (Poison, Irritant, Etc.)
LC_{50} Inhalation (rat) 8,000 ppm/7 hours	Inhalation: Toxic
LD_{50} Dermal (rabbit) 15g/kg (2)	Skin/Eye: Liquid mildly irritating to skin; eye irritant
LD_{50} Ingestion (rat) 10-12g/kg (See Section 5)	Ingestion: Not significantly toxic
Fish, LC_{50} (Lethal Concentration) Not Determined	Aquatic:

Human Exposure Information/Data:

24-HOUR EMERGENCY ASSISTANCE: (313) 358-5800

SECTION 5 · EFFECTS OF OVEREXPOSURE

This section covers effects of overexposure for inhalation, eye/skin contact, ingestion and other types of overexposure information in the order of the most hazardous and the most likely route of overexposure.

missible Exposure Limits (TLV):

350 ppm - 8-hour time-weighted average (TWA) - OSHA 29CFR 1910.1000 (May 28, 1975).

Acute

Primarily a central nervous system depressant. Inhalation can cause irritation of the respiratory system, dizziness, nausea, lightheadedness, headache, loss of coordination and equilibrium, unconsciousness and, if exposed to high concentrations in confined or poorly ventilated areas, even death. Depression of the circulatory system has been reported as a result of overexposure to 1,1,1-trichloroethane. The heart may be sensitized by overexposure and ventricular arrhythmia may be induced by epinephrine administration.

Liquid splashed in the eyes can result in disconfort, pain and irritation. Prolonged or repeated contact with liquid on the skin can cause irritation and dermatitis. The problem may be accentuated by liquid becoming trapped against the skin by contaminated clothing and shoes. Skin absorption can occur.

Chronic

olonged exposure above the OSHA permissible exposure limits may result in liver and dney damage. 1,1,1-trichloroethane has been extensively studied for cancer both in the U.S. and Europe by government, industry and academia. There is no documented evidence that 1,1,1-trichloroethane causes an increased cancer incidence in humans.

EMERGENCY AND FIRST AID PROCEDURES:

Inhalation: Remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician.

Eye or Skin Contact: Flush eyes and skin with plenty of water (soap and water for skin) for at least 15 minutes, while removing contaminated clothing and shoes. If irritation occurs, consult a physician.

Ingestion: If conscious, drink large quantities of water, DO NOT induce vomiting. Take immediately to a hospital or physician. If unconscious, or in convulsions, take immediately to a hospital. DO NOT give anything by mouth to an unconscious person.

Notes to Physician (Including Antidotes): NEVER administer adrenalin following 1,1,1-trichloroethane overexposure. Increased sensitivity of the heart to adrenalin may be caused by overexposure to 1,1,1-trichloroethane.

SECTION 6 · REACTIVITY DATA

Stability: Stable	**Conditions to Avoid:** Avoid open flames, hot glowing surfaces or electric arcs.
Hazardous Polymerization: Will not occur.	**Conditions to Avoid:** None

Incompatibility (Materials to Avoid):
Avoid contamination with caustic soda, caustic potash or oxidizing materials. Shock sensitive explosives may be formed. Avoid contact with aluminum, magnesium, zinc and alloys thereof under high pressures. See Detrex warning letter Form SoL 8208.21 attached.

Hazardous Decomposition Products: Hydrogen chloride and possibly traces of phosgene.

SECTION 7 · SPILL OR LEAK PROCEDURES (See Detrex Forms SoL 8208.14 and SoL 8208.15 attached)

Steps to be Taken if Material is Spilled or Released: Immediately evacuate the area and provide maximum ventilation. Unprotected personnel should move upwind of spill. Only personnel equipped with proper respiratory and skin/eye protection should be permitted in area. Dike area to contain spill. Take precautions as necessary to prevent contamination of ground and surface waters. Recover or absorb spilled material on sawdust or vermiculite and sweep into closed containers for disposal. After all visible traces have been removed, thoroughly wet vacuum the area. DO NOT flush to sewer. If area of spill is porous, remove as much contaminated earth and gravel, etc., as necessary and place in closed containers for disposal. (See Below)

Waste Disposal Method: Contaminated sawdust, vermiculite or porous surface must be disposed of in a permitted hazardous waste management facility. Recovered liquids may be re-processed or incinerated or must be treated in a permitted hazardous waste management facility. Care must be taken when using or disposing of chemical materials and/or their containers to prevent environmental contamination. It is your duty to dispose of the chemical materials and/or their containers in accordance with the Clean Air Act, the Clean Water Act, the Resource Conservation and Recovery Act as well as any other relevant federal, state or local laws/regulations regarding disposal.

SECTION 8 · SPECIAL PROTECTION INFORMATION

Respiratory Protection: For emergencies or working in confined areas, wear self-contained breathing apparatus or supplied air respiratory protection (use the "buddy system" and ear a safety harness with a lifeline). In other circumstances involving potential erexposure, use NIOSH/MSHA-approved organic vapor respirator. (Observe limitations irected by manufacturer.) Respiratory protection program must be in accordance with 29CFR 1910.134.

Ventilation (Type): Dilution (General) or Local Exhaust - Sufficient to maintain workplace concentration below permissible exposure limits.

Eye Protection: Splashproof Goggles

Gloves: polyethylene, neoprene or polyvinyl alcohol

Other Protective Equipment: Safety shower and eye-wash fountain in immediate area. Personnel protective clothing and use of equipment must be in accordance with 29CFR 1910.133 and 29CFR 1910.132.

SECTION 9 · SPECIAL PRECAUTIONS

Precautions to be Taken During Handling and Storing:

- Do not use in poorly ventilated or confined areas.
- 1,1,1-trichloroethane vapors are heavier than air and will collect in low areas.
- Keep container closed when not in use.
- Do not store in open, unlabeled or mislabeled containers.
- Liquid oxygen or other strong oxidants may form explosive mixtures with 1,1,1-trichloroethane.
- This material or its vapors when in contact with flames, hot glowing surfaces or electric arcs can decompose to form hydrogen chloride gas and traces of phosgene.
- AVOID CONTAMINATION OF WATER SUPPLIES: Handling, storage, and use procedures must be carefully monitored to avoid spills or leaks. Any spill or leak has the potential to cause underground water contamination which may, if sufficiently severe, render a drinking water source unfit for human consumption. Contamination that does occur cannot e easily corrected.
  chlorinated solvent used as a flashpoint suppressant must be added in sufficient quantity or the resultant mixture may have a flashpoint lower than the flammable component.
- Caution should be taken not to use in pressurized or totally enclosed system of light metal construction such as aluminum, magnesium, zinc or alloys thereof. Example, paint or adhesive spray system. (See Detrex Form SoL 8208.21 attached.)

Other Precautions:

- AVOID PROLONGED OR REPEATED BREATHING OF VAPORS. High vapor concentrations can cause dizziness, unconsciousness or death. Long-term overexposure may cause liver/kidney injury.
- USE ONLY WITH ADEQUATE VENTILATION. Ventilation must be sufficient to limit employee exposure to 1,1,1-trichloroethane below OSHA permissible limits (8-hour TWA 350 ppm). Observance of lower limits (outlined in Section 4) is advisable.
- AVOID CONTACT WITH EYES. Will cause irritation and pain.
- AVOID PROLONGED OR REPEATED CONTACT WITH SKIN. May cause irritation or dermatitis.
- DO NOT TAKE INTERNALLY. Swallowing may cause injury or death.
- DO NOT EAT, DRINK, OR SMOKE IN WORK AREAS.

References:

1. NIOSH Registry of Toxic Effects of Chemical Substances, 1978
2. Industrial Hygiene and Toxicology, Volume II, Second Edition, F.A. Patty, 1963
3. Dangerous Properties of Industrial Materials, Fifth Edition, N.I. Sax, 1979
4. Industrial Toxicology, Hamilton and Hardy, 1974
5. Toxicity and Metabolisms of Industry Solvents, Browning, 1965
6. Toxicology, the Basic Science of Poisons, Casarett and Doull, 1980
7. Federal Register, 45FR Hazardous Waste Management Systems Part III, Identification and Listing of Hazardous Wastes, Page 33084, May 19,1980
8. EPA Science Advisory Board, Subcommittee on Airborne Carcinogens, September, 1980

omments: As this solvent (1,1,1-trichloroethane) is used to clean and/or degrease a wide variety of metal and plastic parts, it should always be used in conjunction with properly designed and fully controlled degreasing equipment that is in compliance with the U.S. Environmental Protection Agency, OAQPS Guidelines, "Control of Volatile Organic Emissions from Solvent Metal Cleaning", and/or all other applicable federal, state and local regulatory guidelines.

Material Safety Data Sheet

MSD 8208.19

DETREX CHEMICAL INDUSTRIES, INC.
P.O. BOX 501,
DETROIT, MICHIGAN 48232

Approved by U.S. Dept. of Labor as "Essentially similar" to Form OSHA-20

Date: August, 1982	Edition: First
Chemical Name and Synonyms: Trichloroethylene; trichloroethene CAS No.: 79-01-6	Trade Name and Synonyms: PERM-A-CLOR® NA, Trichlor, Trichlorethylene
Chemical Family: Halogenated Hydrocarbons	Formula: $CHCl = CCl_2$
DOT Shipping Name: trichloroethylene	DOT Hazard Class: ORM-A UN1710 (RQ 1000#/454kg)

SECTION 1 · PHYSICAL DATA

Boiling Point @ 760 mm Hg: 188°F	Vapor Density (Air=1): 4.54	Specific Gravity (H_2O=1): (20°/20°C) 1.465	pH of Solutions: 6.7 to 7.5
Freezing/Melting Point: -123.5°F -86.4°C	Solubility (Weight % in Water): 0.11 @ 25°C	Bulk Density: @ 20°C 12.2 lbs./gal.	Volume % Volatile: Essentially 100
Vapor Pressure: @ 20°C = 57.8mmHg	Evaporation Rate (ethyl ether=1): 0.28	Heat of Solution: Not Applicable	Appearance and Odor: Clear, colorless liquid with ether-like odor.

SECTION 2 · HAZARDOUS INGREDIENTS

	%	Hazard Data
Trichloroethylene (Stabilized)	100	See Sections 4 and 5

SECTION 3 · FIRE AND EXPLOSION HAZARD DATA

Flash Point °F (Method Used) None when tested in accordance with DOT requirements. (See Below)	Flammable Limits in Air (% by Volume) See Below LEL: 12.5% UEL: 90%	Extinguishing Media: Water, dry chemicals or carbon dioxide.

Special Fire Fighting Procedures: Fire fighters should wear NIOSH/MSHA pressure-demand, self-contained breathing apparatus for possible exposure to hydrogen chloride and possibly traces of phosgene.

Unusual Fire and Explosion Hazards: Vapors concentrated in a confined or poorly ventilated area can be ignited upon contact with a spark, flame or high-intensity source of heat. This can occur at concentrations of approximately 12.5% and above by volume. Decomposition or burning can produce hydrogen chloride and possibly traces of phosgene.

Also see Detrex warning letter Form SoL 8208.21 attached.

SECTION 4 · HEALTH HAZARD DATA

Permissible Exposure Limits (TLV): See Section 5

Toxicity Data Ref. (1),(2)	Classification (Poison, Irritant, Etc.)
LCLoInhalation (rat) - 8,000 ppm/4 hour	Inhalation: Toxic
LD_{50} Dermal	Skin/Eye: Liquid mildly irritant to skin; eye irritant.
LD_{50} Ingestion (rat) - 4,900 - 7,000 mg/kg	Ingestion: Slightly to moderately toxic
Fish, LC_{50}(Lethal Concentration) Not Determined	Aquatic:

Human Exposure Information/Data: Unconfirmed data[1] exists which indicate that trichloroethylene by ingestion may be more toxic to humans than indicated by the available animal data. Such unconfirmed data report poisonings at doses as low as 50 mg/kg.

24-HOUR EMERGENCY ASSISTANCE: (313) 358-5800

Section 4 (Cont'd) - Permissible Exposure Limits

Current OSHA permissible exposure limits (29CFR 1910.1000) are 100 ppm (8-hour TWA); 100-200 ppm periodic excursions are allowed providing 8-hour TWA is at or below 100 ppm; ^00-300 ppm excursions allowed only for maximum of 5 minutes in any 2-hour period: 0 ppm maximum allowable concentration (must not be exceeded).

SECTION 5 · EFFECTS OF OVEREXPOSURE

is section covers effects of overexposure for inhalation, eye/skin contact, ingestion and other types of overexposure formation in the order of the most hazardous and the most likely route of overexposure.

Acute: Irritant and central nervous system depressant. Inhalation can cause irritation of the respiratory tract, dizziness, nausea, headache, loss of coordination and equilibrium, unconsciousness and, if exposed at high concentrations in confined or poorly ventilated areas, even death. Fatalities following severe acute exposure at high concentrations have been attributed to ventricular fibrillation resulting in cardiac failures.[3]

Liquid splashed in the eye can result in discomfort, pain and irritation. Prolonged or repeated contact with liquid on the skin can cause irritation and dermatitis. The problem may be accentuated by liquid becoming trapped against the skin by contaminated clothing and shoes. Skin absorption can occur.

Chronic: Prolonged exposure above the OSHA permissible exposure limits may result in liver and kidney damage. Trichloroethylene has been extensively studied for cancer both in the U.S. and Europe by government, industry and academia. There is no documented evidence that Trichloroethylene causes an increased cancer incidence in humans.

EMERGENCY AND FIRST AID PROCEDURES:

Inhalation: Remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician .

Eye or Skin Contact: Flush eyes and skin with plenty of water (soap and water for skin) for at least 15 minutes, while removing contaminated clothing and shoes. If irritation occurs, consult a physician.

Ingestion: If conscious, drink a quart of water. DO NOT induce vomiting. Take immediately to a hospital or physician. If unconscious, or in convulsions, take immediately to a hospital or physician. DO NOT give anything by mouth to an unconscious person.

Notes to Physician (Including Antidotes): NEVER administer adrenalin following trichloroethylene overexposure. Increased sensitivity of the heart to adrenalin may be caused by overexposure to trichloroethylene.

SECTION 6 · REACTIVITY DATA

Stability: Stable	**Conditions to Avoid:** Avoid open flames, hot glowing surfaces or electric arcs.
:ardous Polymerization: Will not occur	**Conditions to Avoid:** None

Incompatibility (Materials to Avoid): Avoid contamination with caustic soda. caustic potash or oxidizing materials. Shock sensitive explosives may be formed.
See Detrex warning letter Form SoL 8208.21 attached.

Hazardous Decomposition Products: Hydrogen chloride and possibly traces of phosgene.

SECTION 7 · SPILL OR LEAK PROCEDURES (See Detrex Forms SoL 8208.14 and SoL 8208.15 attached)

Steps to be Taken if Material is Spilled or Released: Immediately evacuate the area and provide maximum ventilation. Unprotected personnel should move upwind of spill. Only personnel equipped with proper respiratory and skin/eye protection should be permitted in area. Dike area to contain spill. Take precautions as necessary to prevent contamination of ground and surface waters. Recover or absorb spilled material on sawdust or vermiculite and sweep into closed containers for disposal. After all visible traces have been removed, thoroughly wet vacuum the area. DO NOT flush to sewer. If area of spill is porous, remove as much contaminated earth and gravel, etc., as necessary and place in closed containers for disposal. (See Below)

Waste Disposal Method: Contaminated sawdust, vermiculite or porous surface must be disposed of in a permitted hazardous waste management facility. Recovered liquids may be reprocessed or incinerated or must be treated in a permitted hazardous waste management facility.[7] Care must be taken when using or disposing of chemical materials and/or their containers to prevent environmental contamination. It is your duty to dispose of the chemical materials and/or their containers in accordance with the Clean Air Act, the Clean Water Act, the Resource Conservation and Recovery Act and all relevant state or local laws/regulations regarding disposal of hazardous waste.

SECTION 8 · SPECIAL PROTECTION INFORMATION

Respiratory Protection: For emergencies or working in confined areas, wear self-contained breathing apparatus or supplied air respiratory protection (use "buddy system", also use harness and lifeline). In other circumstances involving potential overexposures, use OSH/MSHA-approved organic vapor respirator. (Observe limitations directed by manufacturer.) Respiratory protection program must be in accordance with 29CFR 1910.134.

Ventilation (Type): Mechanical (General) - Sufficient to maintain workplace concentration below permissible exposure limits.

Eye Protection: Splashproof goggles.

Gloves: Polyethylene, neoprene or polyvinyl alcohol.

Other Protective Equipment: Safety shower and eye-wash fountain in immediate area. Personnel protective clothing and use of equipment must be in accordance with 29CFR 1910.133 and 29CFR 1910.132.

SECTION 9 · SPECIAL PRECAUTIONS

Precautions to be Taken During Handling and Storing:

Do not use in poorly ventilated or confined spaces.

- Trichloroethylene vapors are heavier than air and will collect in low areas. Keep container closed when not in use.
- Do not store in open, unlabeled or mislabeled containers.
- Liquid oxygen or other strong oxidants may form explosive mixtures with trichloroethylene
- This material or its vapors when in contact with flames, hot glowing surfaces or electric arcs can decompose to form hydrogen chloride gas and traces of phosgene.
- AVOID CONTAMINATION OF WATER SUPPLIES: Handling, storage and use procedures must be carefully monitored to avoid spills or leaks. Any spill or leak has the potential to cause underground water contamination which may, if sufficiently severe, render a drinking water source unfit for human consumption. Contamination that does occur cannot be easily corrected.

Other Precautions:

- AVOID PROLONGED OR REPEATED BREATHING OF VAPORS. High vapor concentrations can cause dizziness, unconsciousness or death. Long term overexposure may cause liver/kidney injury.
- USE ONLY WITH ADEQUATE VENTILATION. Ventilation must be sufficient to limit employee exposure to trichloroethylene in work area at or below OSHA permissible exposure limits (8-hour TWA - 100 ppm; ceiling - 200 ppm; maximum peak - 300 ppm, 5 minutes in every 2 hours). Observance of lower limits (outlined in Section 4) is advisable.
- AVOID CONTACT WITH EYES. Will cause irritation and pain.
- AVOID PROLONGED OR REPEATED CONTACT WITH SKIN. May cause irritation or dermatitis.
- DO NOT TAKE INTERNALLY. Swallowing may cause injury or death.
- DO NOT EAT, DRINK OR SMOKE IN WORK AREAS.

References:

1. NIOSH Registry of Toxic Effects of Chemical Substances, 1978
2. Industrial Hygiene and Toxicology, Volume II, Second Edition, F. A. Patty, 1963
3. Dangerous Properties of Industrial Materials, Fifth Edition, N. I. Sax, 1979
4. Industrial Toxicology, Hamilton and Hardy, 1974
5. Toxicity and Matabolisms of Industrial Solvents, Browning, 1965
6. Toxicology, the Basic Science of Poisons, Casarett and Doull, 1975
7. Federal Register, 45FR Hazardous Waste Management Systems Part III, Identification and Listing of Hazardous Wastes, Page 33084, May 19, 1980
8. EPA Science Advisory Board, Subcommittee on Airborne Carcinogens, September, 1980

Comments:

As this solvent (trichloroethylene) is used primarily to clean and/or degrease a wide variety of metal and plastic parts, it should always be used in conjunction with properly designed and fully controlled solvent vapor degreasing equipment that is in compliance with the U.S. Environmental Protection Agency, OAQPS Guidelines, "Control of Volatile Organic Emissions from Solvent Metal Cleaning", and/or all other applicable federal, state or local regulatory guidelines.

Material Safety Data Sheet

MSD 8208.22

rev. 11/85

DETREX CORPORATION P.O. Box 5111, Southfield, MI 48086-5111

Approved by U.S. Dept. of Labor as "Essentially similar" to Form OSHA-20

Date: November, 1985	Edition: Second
Chemical Name and Synonyms: Perchloroethylene; tetrachloroethylene CAS No.: 127-18-4	Trade Name and Synonyms: DETREX PERK GOLD SHIELD PERK Perchlor, Perchloroethylene
Chemical Family: Halogenated Hydrocarbons	Formula: $CCl_2=CCl_2$
DOT Shipping Name: Tetrachloroethylene	DOT Hazard Class: ORM-A UN1897

SECTION 1 · PHYSICAL DATA

Boiling Point @ 760 mm Hg. 121°C 250°F	Vapor Density (Air=1). 5.83	Specific Gravity (H_2O=1): 1.6 @ 20/20°C	pH of Solutions 6.8 to 8.4
Freezing/Melting Point: -22.3°C -8.2F	Solubility (Weight % in Water): 0.015% @ 25°C	Bulk Density: 13.6 lbs./gal. @ 20°C	Volume % Volatile: 100
Vapor Pressure: 14.2mmHg @ 20°C	Evaporation Rate (ethyl ether-1):0.09	Heat of Solution: Not Applicable	Appearance and Odor: Clear, colorless liquid with ether-like odor.

SECTION 2 · HAZARDOUS INGREDIENTS

	%	Hazard Data
Perchloroethylene (Stabilized)	100	See Section 4 & 5

SECTION 3 · FIRE AND EXPLOSION HAZARD DATA

Flash Point °F (Method Used) None when tested in accordance with DOT requirements	Flammable Limits in Air (% by Volume) LEL: none UEL: none	Extinguishing Media: For fires involving perchloroethylene, use water, dry chemical or carbon dioxide.

Special Fire Fighting Procedures: Fire fighters should wear NIOSH/MSHA-approved pressure-demand self-contained breathing apparatus for possible exposure to hydrogen chloride and possible traces of phosgene.

Unusual Fire and Explosion Hazards: Perchloroethylene involved in fires can decompose to hydrogen chloride and possible traces of phosgene.

SECTION 4 · HEALTH HAZARD DATA

Toxicity Data See Reference 1	Classification (Poison, Irritant, Etc.)
LC_{50} Inhalation (rat) 4,000 ppm (4 hours)	Inhalation: Moderately Toxic
LD_{50} Dermal Not Determined	Skin Not Determined
Skin/Eye Irritation See Section 5	Skin/Eye: Mildly irritating to skin; eye irritant
LD_{50} Ingestion (rabbit) 5000 mg/kg	Ingestion: Slightly Toxic
Fish, LC_{50} (Lethal Concentration) 96hr. TLM 100-10 ppm	Aquatic: Toxic

Human Exposure Information/Data: Unconfirmed data[1] exists which indicate that perchloroethylene by ingestion may be more toxic to humans than indicated by the available data. Such unconfirmed data report poisonings at doses as low as 500 mg/kg.

24-HOUR EMERGENCY ASSISTANCE: (313) 358-5800

SECTION 5 · EFFECTS OF OVEREXPOSURE

This section covers effects of overexposure for inhalation, eye/skin contact, ingestion and other types of overexposure information in the order of the most hazardous and the most likely route of overexposure.

P ıssible Exposure Limits The individual permissible exposure limits are as follows:

OSHA: 100 ppm, 8-hour TWA (time-weighted average); 100-200 ppm periodic excursions are allowed providing 8-hour TWA is at or below 100 ppm; 200-300 ppm excursions allowed only for maximum of 5 minutes in any 3-hour period; 300 ppm maximum allowable concentration (must not be exceeded); 29CFR 1910.1000.

ACGIH: 50 ppm, 8-hour TWA (time-weighted average); 200 ppm, STEL (15-minute short-term exposure limit)

ACUTE

Inhalation: Perchloroethylene is primarily a central nervous system depressant and can cause possible central nervous system damage with overexposure. Inhalation can cause irritation of the respiratory tract, dizziness, nausea, headache, loss of coordination and equilibrium, unconsciousness and even death in confined or poorly ventilated area. Fatalities following severe acute exposure to various chlorinated solvents have been attributed to ventricular fibrillation.

Eye/Skin: Liquid splashed in the eye can result in discomfort, pain and irritation. Prolonged or repeated contact with liquid on the skin can cause irritation and dermatitis. The problem may be accentuated by liquid becoming trapped against the skin by contaminated clothing and shoes, and skin absorption can occur.

Ingestion: Swallowing of this material may result in irritation of the mouth and GI t along with other effects as listed above for Inhalation. Vomiting and subsequent iration into the lungs may lead to chemical pneumonia and pulmonary edema which is a potentially fatal condition.

CHRONIC

Prolonged exposure above the OSHA permissible exposure limits may result in liver and kidney damage. Perchloroethylene has been extensively studied for its cancer potential. There is no documented evidence to suggest that perchloroethylene causes an increased cancer incidence in humans, nor does available information from animal studies suggest that this material poses a carcinogenic risk to humans under conditions adhering to recommended occupational guidelines.

EMERGENCY AND FIRST AID PROCEDURES:

Inhalation: Remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician.

Eye or Skin Contact: Flush eyes and skin with plenty of water (soap and water for skin) for at least 15 minutes, while removing contaminated clothing and shoes. If irritation occurs, consult a physician. Thoroughly clean contaminated clothing and shoes before reuse or discard.

Ingestion: If conscious, drink large quantities of water. DO NOT induce vomiting. Take immediately to a hospital or physician. If unconscious, or in convulsions, take immediately to a hospital. DO NOT attempt to give anything by mouth to an unconscious person.

Notes to Physician (Including Antidotes): NEVER administer adrenalin following perchloroethylene overexposure. Increased sensitivity of the heart to adrenalin may be caused by overexposure to perchloroethylene.

SECTION 6 · REACTIVITY DATA

Stability: Stable	**Conditions to Avoid:** Avoid open flames, hot glowing surfaces or electric arcs.
Hazardous Polymerization: Will not occur	**Conditions to Avoid:** None

Incompatibility (Materials to Avoid): Avoid contamination with caustic soda, caustic potash or oxidizing materials. Shock sensitive explosives may be formed.

Hazardous Decomposition Products: Hydrogen chloride and possibly traces of phosgene.

SECTION 7 · SPILL OR LEAK PROCEDURES

Steps to be Taken if Material is Spilled or Released: Immediately evacuate the area and provide maximum ventilation. Unprotected personnel should move upwind of spill. Only personnel equipped with proper respiratory and skin/eye protection (See Section 8) should be permitted in area. Dike area to contain spill. Take precautions as necessary to prevent contamination of ground and surface waters. Recover spilled material on adsorbents, such as sawdust or vermiculite, and sweep into closed containers for disposal. After all visible traces, including ignitable vapors, have been removed, thoroughly wet vacuum the area. DO NOT flush to sewer. If area of spill is porous, remove as much earth and gravel, etc. as necessary and place in closed containers for disposal.

Waste Disposal Method: Contaminated sawdust, vermiculite or porous surface must be disposed of in a permitted hazardous waste management facility. Recovered liquids may be reprocessed or incinerated or must be treated in a permitted hazardous waste management facility. Care must be taken when using or disposing of chemical materials and/or their containers in accordance with the Clean Air Act, the Clean Water Act, the Resource Conservation and Recovery Act as well as any other relevant federal, state, or local laws/regulations regarding disposal.

CRA - Hazardous Waste Number - U-210

SECTION 8 · SPECIAL PROTECTION INFORMATION

Respiratory Protection: Use a half or full facepiece organic vapor chemical cartridge or canister respirator when concentrations exceed permissible limits. Use self-contained breathing apparatus (SCBA) or full facepiece airline respirator with auxiliary SCBA operated in the pressure-demand mode emergencies and for all work performed in storage vessels, poorly ventilated rooms, and other fined areas. Respirators must be approved by NIOSH or MSHA. The respirator use limitations made by NIOSH/MSHS[6,7] and by the manufacturer must be observed. Respiratory protection programs must be observed. Respiratory protection programs must be in accordance with 29CFR 1910.134.

Ventilation (Type): Use local exhaust or dilution ventilation as appropriate to control exposures to below permissible limits.

Eye Protection: Splashproof goggles

Gloves: Viton , Polyvinyl alcohol*.
For limited service only: Nitrile.
(*degrades in water)

Other Protective Equipment: Boots, aprons, or chemical suits should be used when necessary to prevent skin contact. Personnel protective clothing and use of equipment must be in accordance with 29CFR 1910.133 and 29CFR 1910.132.

SECTION 9 · SPECIAL PRECAUTIONS

Precautions to be Taken During Handling and Storing:

- Do not use in poorly ventilated or confined spaces without proper respiratory protection (See Section 8).
- Perchloroethylene vapors are heavier than air and will collect in low areas.
- Keep container closed when not in use.
- Store only in closed, properly labeled containers.
- This material or its vapors when in contact with flames, hot glowing surfaces or electric arcs can decompose to form hydrogen chloride gas and traces of phosgene.
- AVOID CONTAMINATION OF WATER SUPPLIES. Handling, storage and use procedures must be carefully monitored to avoid spills or leaks. Any spill or leak has the potential to cause underground water contamination which may, if sufficiently severe, render a drinking water source unfit for human consumption. Contamination that does occur cannot be easily corrected.
  A chlorinated solvent used as a flashpoint suppressant must be added in sufficient quantity or the resultant mixture may have a flashpoint lower than the flammable component.
- Do not use cutting or welding torches on drums that contained perchloroethylene unless properly purged and cleaned.

Other Precautions:

- Do not breathe vapors. High vapor concentrations can cause dizziness, unconsciousness, possible central nervous system damage or death. Long-term overexposure may cause liver/kidney injury.
- Use only with adequate ventilation. Ventilation must be sufficient to limit employee exposure to perchloroethylene below permissible exposure limits. Observance of lower limits (outlined in Section 5) is advisable.
- Avoid contact with eyes. Will cause irritation and pain
- Avoid prolonged or repeated contact with skin. May cause irritation or dermatitis.
- Do not swallow. Swallowing may cause injury or death.
- Do not eat, drink, or smoke in work areas.

References:

1. NIOSH registry of Toxic Effects of Chemical Substances, 1978
2. Industrial Hygiene and Toxicology, Volume II, Second Edition, F. A. Patty, 1963
3. Dangerous Properties of Industrial Materials, Fifth Edition, N. I. Sax, 1979
4. EPA Science Advisory Board, Subcommittee on Airborne Carcinogens, September, 1980
5. Encyclopedia of Chemical Technology, Volume 5, Third Edition, Kirk-Othmer, 1979
6. NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, DHHS (NIOSH) Publication No. 81-123, January, 1981
7. NIOSH/OSHA Pocket Guide to Chemical Hazards, DHEW (NIOSH) Publication No. 78-210 September, 1978

Comments: As this solvent (perchloroethylene) is used to clean and/or degrease a wide variety of metal and plastic parts, it should always be used in conjunction with properly designed and fully controlled solvent vapor degreasing equipment that is in compliance with the U. S. Environmental Protection Agency, OAQPS Guidelines, "Control of Volatile Organic Emissions from Solvent Metal Cleaning", and/or any other applicable federal, state or local regulatory guidelines.

Perchloroethylene is also used extensively in the commercial drycleaning of clothes. It uld always be used in conjunction with properly designed and fully controlled equipment that is compliance with all applicable federal, state and local regulatory guidelines.

DETREX AUTHORIZED SIGNATURE James R. Goodrich

Material Safety Data Sheet

MSD 8208.23

DETREX CHEMICAL INDUSTRIES, INC.
P.O. BOX 501,
DETROIT, MICHIGAN 48232

Approved by U.S. Dept. of Labor as "Essentially similar" to Form OSHA-20

Date:	Edition:
Chemical Name and Synonyms: Methylene Chloride; dichloromethane CAS No.: 75-09-2	Trade Name and Synonyms: Methylene Chloride
Chemical Family: Halogenated Hydrocarbons	Formula: CH_2Cl_2
DOT Shipping Name: Methylene Chloride	DOT Hazard Class: ORM-A - UN 1593

SECTION 1 · PHYSICAL DATA

Boiling Point @ 760 mm Hg:	Vapor Density (Air=1):	Specific Gravity (H_2O=1):	pH of Solutions:
103.6°F (39.8°C)	@ 20°C = 2.93	1.32	Neutral
Freezing/Melting Point:	**Solubility (Weight % in Water):**	**Bulk Density:**	**Volume % Volatile:**
-142.1°F (-96.7°C)	2g/100 ml	@ 20°C 11.15 lbs./gal.	Essentially 100
Vapor Pressure:	**Evaporation Rate**	**Heat of Solution:**	**Appearance and Odor:**
@ 20°C = 349 mmHg	(ethyl ether = 1): 0.71	Not Applicable	Clear, colorless liquid with ether-like odor.

SECTION 2 · HAZARDOUS INGREDIENTS

	%	Hazard Data
Methylene Chloride (Stabilized)	100	See Sections 4 and 5

SECTION 3 · FIRE AND EXPLOSION HAZARD DATA

Flash Point °F (Method Used)	Flammable Limits in Air (% by Volume)	Extinguishing Media:
None when tested in accordance with DOT requirements.	See Below LEL: 12% UEL: 19%	For fires involving methylene chloride, use water, dry chemicals or CO_2.

Special Fire Fighting Procedures: Fire fighters should wear NIOSH/MSHA-approved, self-contained breathing apparatus for possible exposure to hydrogen chloride and possible traces of phosgene.

Unusual Fire and Explosion Hazards: Vapors concentrated in a poorly ventilated area can be ignited upon contact with a spark, flame or high intensity source of heat. This can occur at concentrations between 12% and 19% by volume. Decomposition or burning can produce hydrogen chloride and possibly traces of phosgene. Also see Detrex warning letter form SoL 8208.21 attached.

SECTION 4 · HEALTH HAZARD DATA

Permissible Exposure Limits (TLV): 500 ppm (TWA) See Section 4 (Cont'd) next page

Toxicity Data	Classification (Poison, Irritant, Etc.)
LC_{50} Inhalation: LCLo (guinea pig) 5,000ppm/2hrs. TCLo*(human) 500ppm/8hrs.	Inhalation: Toxic
LD_{50} Dermal	Skin/Eye: Liquid mildly irritating to skin; eye irritant.
LD_{50} Ingestion - (rat) = 2,136mg/kg	Ingestion: Slightly toxic
Fish, LC_{50} (Lethal Concentration) Not determined	Aquatic:

Human Exposure Information/Data:

* Lowest published toxic concentration.

24-HOUR EMERGENCY ASSISTANCE: (313) 358-5800

Section 4 (cont'd) - Permissible Exposure Limit

Current OSHA permissible exposure limits (29CFR 1910.1000) are 500 ppm (8-hour TWA); 500-1,000 ppm period excursions are allowed providing TWA is at or below 500ppm; 1,000-2,000ppm excursions allowed only for maximum of five minutes in any 2-hour period -- 2,000ppm maximum allowable concentration (must not be exceeded).

*NIOSH recommends that the TWA exposure limit for methylene chloride is 75ppm. In the absence of occupational exposure to carbon monoxide (CO) above a TWA of 9ppm up to a 10-hour workday, occupational exposure to methylene chloride shall be controlled so that workers are not exposed to methylene chloride in excess of 75ppm (261 mg/cu m) determined as a TWA for up to a 10-hour workday, 40-hour workweek. In the presence of exposure to CO in the work environment at more than 9ppm as a TWA for up to a 10-hour workday, exposure limits of CO or methylene or both shall be reduced to satisfy the relationship:

$$\frac{C(CO)}{L(CO)} + \frac{C(CH_2Cl_2)}{L(CH_2Cl_2)} \leq 1$$

where: C(CO) = TWA exposure concentration of CO, ppm

L(CO) = the recommended TWA exposure limit of CO = 35 ppm

$C(CH_2Cl_2)$ = TWA exposure concentration of methylene chloride, ppm

$L(CH_2Cl_2)$ = the recommended TWA exposure limit of methylene chloride = 75 ppm

Occupational exposure shall be controlled so that workers are not exposed to methylene chloride above a peak concentration of 500ppm (1,740 mg/cu m) as determined by a 15-minute sampling period.

Employees working with methylene chloride should be aware of this hazard. This toxic effect is "additive" in nature with the risk being greater for smokers, who generally have higher levels of carboxyhemoglobin. Employees with a history of cardiovascular disease should not be allowed to work with methylene chloide unless approved by a physician.

SECTION 5 · EFFECTS OF OVEREXPOSURE

This section covers effects of overexposure for inhalation, eye/skin contact, ingestion and other types of overexposure information in the order of the most hazardous and the most likely route of overexposure.

Effects of Overexposure [2]

Acute: Inhalation effect is primarily narcosis. Principal symptoms may be headache, dizziness, nausea, tingling or numbness of the extremities, senses of fullness in the head, sense of warmth, stupor or dullness, lethargy and drunkeness. Exposure to very high concentrations may lead to unconsciousness or even death in confined or poorly ventilated areas.

Chronic: Several chronic inhalation studies reported by NIOSH revealed that test animals exposed to methylene chloride concentrations as high as 10,000ppm, showed slight liver and kidney changes. The results of these studies indicate that prolonged exposure limits may result in liver and kidney damage.

Chronic inhalation studies, cosponsored by several methylene chloride producers, were recently completed on rats. The results of these studies were reported to have revealed a mathematically significant increase in malignant salivary gland tumors in the group of male rats from the study's highest exposure level (3,500ppm).

Liquid splashed in the eyes can result in discomfort, pain and irritation. Prolonged or repeated contact with liquid on the skin can cause irritation and dermatitis. The problem may be accentuated by liquid becoming trapped against the skin by contaminated clothing and shoes. Skin absorption can occur.

Research has recently shown that methylene chloride is metabolized by the body to carbon monoxide.[4] Further, the amount of carbon monoxide formed is directly related to the amount of methylene chloride absorbed and can be sufficient to produce a substantial stress on the cardiovascular system through the elevation of the level of carboxyhemoglobin (COHb) -- the product formed by the combination of carbon monoxide and the blood's hemoglobin thus effectively reducing the amount of hemoglobin available for the transport of oxygen throughout the body.

EMERGENCY AND FIRST AID PROCEDURES:

Inhalation: Remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician.

Eye or Skin Contact: Flush eyes and skin with plenty of water (soap and water for skin) for at least 15 minutes, while removing contaminated clothing and shoes. If irritation occurs, consult a physician.

Ingestion: If conscious, drink a quart of water. DO NOT induce vomiting. Take immediately to a hospital or physician. If unconscious, or in convulsions, take immediately to a hospital or physician. DO NOT give anything by mouth to an unconscious person.

Notes to Physician (Including Antidotes): NEVER administer adrenalin following methylene chloride overexposure. Increased sensitivity of the heart to adrenalin may be caused by overexposure to methylene chloride.

SECTION 6 · REACTIVITY DATA

Stability: Stable	**Conditions to Avoid:** Avoid open flames, hot glowing surfaces or electric arcs.
Hazardous Polymerization: Will not occur	**Conditions to Avoid:** None

Incompatibility (Materials to Avoid): Avoid contamination with caustic soda, caustic potash or oxidizing materials. Shock sensitive explosives may be formed. Avoid contact with aluminum, magnesium, zinc and alloys thereof under high pressures. See Detrex warning letter Form SoL 8208.21 attached.

Hazardous Decomposition Products: Hydrogen chloride and possibly traces of phosgene.

SECTION 7 · SPILL OR LEAK PROCEDURES (See Detrex Forms SoL 8208.14 and SoL 8208.15 attached)

Steps to be Taken if Material is Spilled or Released: Immediately evacuate the area and provide maximum ventilation. Unprotected personnel should move upwind of spill. Only personnel equipped with proper respiratory and skin/eye protection should be permitted in area. Dike area to contain spill. Take precautions as necessary to prevent contamination of ground and surface waters. Recover or absorb spilled material on sawdust or vermiculite and sweep into closed containers for disposal. After all visible traces have been removed, thoroughly wet vacuum the area. DO NOT flush to sewer. If area of spill is porous, remove as much contaminated earth and gravel, etc., as necessary and place in closed containers for disposal. (See Below).

Waste Disposal Method: Contaminated sawdust, vermiculite or porous surface must be disposed of in a permitted hazardous waste management facility. Recovered liquids may be reprocessed or incinerated or must be treated in a permitted hazardous waste management facility. Care must be taken when using or disposing of chemical materials and/or their containers to prevent environmental contamination. It is your duty to dispose of the chemical materials and/or their containers in accordance with the Clean Air Act, the Clean Water Act, the Resource Conservation and Recovery Act and all relevant state or local laws/regulations regarding disposal.

SECTION 8 · SPECIAL PROTECTION INFORMATION

Respiratory Protection: For emergencies or working in confined areas, wear self-contained breathing apparatus or supplied air respiratory protection. (Use the "buddy system" and wear a safety harness with lifeline). In other circumstances involving potentional overexposures, use NIOSH/MSHA-approved organic vapor respirator. (Observe limitations directed by manufacturer.) Respiratory protection program must be in accordance with 29CFR 1910.134.

Ventilation (Type): Mechanical (General) - Sufficient to maintain workplace concentration below permissible exposure limits.

Eye Protection: Splashproof goggles

Gloves: Polyethylene, neoprene or polyvinyl alcohol.

Other Protective Equipment: Safety shower and eye-wash fountain in immediate area. Personnel protective clothing and use of equipment must be in accordance with 29CFR 1910.133 and 29CFR 1910.132.

SECTION 9 · SPECIAL PRECAUTIONS

Precautions to be Taken During Handling and Storing:

- Do not use in poorly ventilated or confined spaces.
- Methylene chloride vapors are heavier than air and will collect in low areas.
- Keep container closed when not in use.
- Do not store in open, unlabeled or mislabeled containers.
- Liquid oxygen or other strong oxidants may form explosive mixtures with methylene chloride.
- This material or its vapors when in contact with flames, hot glowing surfaces or electric arcs can decompose to form hydrogen chloride gas and traces of phosgene.
- AVOID CONTAMINATION OF WATER SUPPLIES: Handling, storage, and use procedures must be carefully monitored to avoid spills or leaks. Any spill or leak has the potential to cause underground water contamination which may, if sufficiently severe, render a drinking water source unfit for human consumption. Contamination that does occur cannot be easily corrected.
- Caution should be taken not use in pressurized or totally enclosed system of light metal construction such as aluminum, magnesium, zinc or alloys thereof. Example, paint or adhesive spray system. (See form Sol. 8208.21 attached.)

Other Precautions:

AVOID PROLONGED OR REPEATED BREATHING OF VAPORS. High vapor concentrations can cause dizziness, unconsciousness or death. Long -term overexposure may cause liver/kidney damage.

- USE ONLY WITH ADEQUATE VENTILATION. Ventilation must be sufficient to limit employee exposure to methylene chloride below OSHA permissible exposure limits (8-hour TWA - 500ppm; ceiling - 1,000ppm; maximum peak - 2,000ppm, 5 minutes in any 2 hours).
- AVOID CONTACT WITH EYES. Will cause irritation and pain.
- AVOID PROLONGED OR REPEATED CONTACT WITH SKIN. May cause irritation or dermatitis.
- DO NOT TAKE INTERNALLY. Swallowing may cause injury or death.

References:

1. NIOSH Registry of Toxic Effects of Chemical Substances, 1978
2. Industrial Hygiene and Toxicology, Volume II, Second Edition, F.A. Patty, 1963
3. Dangerous Properties of Industrial Materials, Fifth Edition, N.I. Sax, 1979
4. Industrial Toxicology, Hamilton and Hardy, 1974
5. Toxicity and Metabolisms of Industrial Solvents, Browning, 1965
6. Toxicology, the Basic Science of Poisons, Casarett and Doull, 1975
7. Federal Register, 45FR Hazardous Waste Management Systems Part III, Identification and Listing of Hazardous Wastes, Page 33084, May 19, 1980
8. EPA Science Advisory Board, Subcommittee on Airborne Carcinogens, September, 1980

Comments: As this solvent (methylene chloride) is used to clean and/or degrease a wide variety of metal and plastic parts, it should always be used in conjunction with properly designed and fully controlled solvent vapor degreasing equipment that is in compliance with the U.S. Environmental Protection Agency OAQPS Guidelines, "Control of Volatile Organic Emissions from Solvent Metal Cleaning", and/or all other applicable federal, state or local regulatory guidelines.

Methylene chloride is also used extensively in industry as a solvent, thinner, paint stripper, etc. It should always be used conjunction with properly designed and fully controlled equipment that is in compliance with applicable federal, state and al regulatory guidelines.

DU PONT

MATERIAL SAFETY DATA SHEET

IDENTIFICATION

Name:
Freon® TF Solvent
Freon® PCA

Chemical Family:
Halogenated Hydrocarbon

Synonyms:
Trichlorotrifluoroethane
R-113, FC-113

Formula:
CCl_2FCClF_2

CAS Name:
Ethane, 1,1,2-Trichloro-1,2,2-Trifluoro

CAS Registry No.
76-13-1

Manufacturer/Distributor:
E. I. du Pont de Nemours & Co. (Inc.)

Medical Emergency Phone:
(800) 441-3637

Address:
Freon® Products Division
Wilmington, DE 19898

Transportation Emergency Phone:
CHEMTREC (800) 424-9300

PHYSICAL DATA

Boiling Point(°F):	117.6	Percent Volatile by Volume:	100%
Density:	1.57 g/cc @/77°F	Vapor Pressure:	334mm Hg @/77°F
Vapor Density (Air = 1):	6.5	Solubility in H_2O:	0.02% by wt. @ 77°F
pH Information:	Neutral	Evaporation Rate (CCl_4 = 1):	0.1
Form: Liquid		Appearance:	Clear
Color: Colorless		Odor:	Slight Ethereal Odor

HAZARDOUS COMPONENTS

Material(s):	Approximate % :
Trichlorotrifluoroethane	100

HAZARDOUS REACTIVITY

Stability:
Material is stable. However, avoid open flames and high temperatures.

[illegible]

Incompatibility:
Alkali or alkaline earth metals - powdered Al, Zn, Be, etc.

Decomposition:
This compound can be decomposed by high temperatures (open flames, glowing metal surfaces, etc.) forming hydrochloric and hydrofluoric acids - possible carbonyl halides.

Polymerization:
Will not occur

FIRE AND EXPLOSION DATA

Flash Point:
None

Method:
TOC

Autoignition Temperature:
Not Determined

Flammable Limits in Air, % by Vol.
Lower: Nonflammable
Upper: Nonflammable

Autodecomposition Temperature:
Not Determined

Fire and Explosion:
Drums may rupture under fire conditions. Decomposition may occur.

Extinguishing Media:
Nonflammable

Special Fire Fighting Instructions:
Self-contained breathing apparatus (SCBA) may be required if drums rupture and contents are spilled under fire conditions.

HEALTH HAZARD INFORMATION

Principal Health Hazards:

Inhalation: Vapor is heavier than air and can cause suffocation by reducing oxygen available for breathing. Breathing high concentrations of vapor may cause light-headedness, giddiness, shortness of breath, and may lead to narcosis, cardiac irregularities, unconsciousness or death. LC 50 Rats 52,000 ppm/4 hrs.

Note: In screening studies with experimental animals, exposure at approximately 5000 ppm (v/v) and above, followed by a large intravenous epinephrine challenge, has induced serious cardiac irregularities.

Skin: Not a corrosive or irritant after single contact; however, repeated liquid contact can cause defatting of the skin resulting in irritation. This material is poorly absorbed through the skin (Rabbit ALD >11,000 mg/kg).

Eye: Liquid contact can cause discomfort, usually no extended effect.

Oral: Although oral toxicity is low [LD 50 Rat 43000 mg/kg], ingestion of FC-113 is to be avoided.

Exposure Limits:

PEL (OSHA)	1,000 ppm
TLV® TWA (ACGIH)	1,000 ppm

Safety Precautions:
Avoid breathing vapors and prolonged skin exposure. Use only in well ventilated area.

First Aid:

Inhalation: Remove to fresh air, call a physician. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Do not give epinephrine or similar drugs.

Note to Physician: Because of a possible increased risk of eliciting cardiac dysrythmias, catecholamine drugs, such as epinephrine, should be considered only as a last resort in life threatening emergencies.

Eye: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Call a physician.

Skin: Flush with water. Get medical attention if irritation is present.

Oral: No specific intervention is indicated as the compound is not likely to be hazardous by ingestion. However, consult a physician if necessary. Do not induce vomiting as the hazard of aspirating the material into the lungs is a greater hazard than allowing it to progress through the intestinal tract.

Medical Conditions Possibly Aggravated by Exposure:

Cardiovascular Disease: See Principal Hazards: Inhalation Section.

Other Health Hazards:

Freon® 113 is not listed as a carcinogen by IARC, NTP or OSHA. Based on animal studies and human experiences this fluorocarbon poses no hazard to man relative to systemic toxicity, carcinogenicity, mutagenicity, or teratogenicity when occupational exposures are below its TLV®.

PROTECTION INFORMATION

Generally Applicable Control Measures:

Normal ventilation for standard manufacturing procedures is generally adequate. Local exhaust should be used when large amounts are released. Mechanical ventilation should be used in low places.

Personal Protective Equipment:

Butyl gloves should be used to avoid prolonged or repeated exposure. Chemical splash goggles should be available for use as needed to prevent eye contact. Under normal manufacturing conditions no respiratory protection is required when using this product. Self-contained breathing apparatus (SCBA) is required if a large spill occurs.

DISPOSAL INFORMATION

Spill, Leak or Release:
Ventilate area. Do not flush into sewers. Dike spill. Collect on absorbent material and transfer to steel drums for recovery or disposal. Comply with federal, state and local regulations on reporting releases.

Waste Disposal: Comply with federal, state and local regulations. Remove to a permitted waste disposal facility. EPA Hazardous Waste Nos. F001 and F002 may apply to waste materials.

SHIPPING INFORMATION

Domestic - Other Than Air (DOT)

Proper Shipping Name	Not Regulated

International Water or Air (IMO/ICAO)

Proper Shipping Name	Not Regulated

Other Information

Shipping Containers	Drums, tank trucks, tank cars
Storage Conditions	Clean, dry area. Do not heat above 125°F.

Date Revised: 10/85
Person responsible: T. D. Armstrong, C&P Dept., Freon® Products Lab, Chestnut Run, Bldg. 711, Wilmington, DE 19898 (302) 999-3847 or (302) 999-4338.

DUPONT

ATTACHMENT C-3

TESTING METHODS

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENTS IN SPENT SOLVENTS BY GAS CHROMATOGRAPHY

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENTS IN SPENT SOLVENTS BY GAS CHROMATOGRAPHY

1.0 SCOPE

1.1 This method is used for the determination of recoverable halogenated and non-halogenated solvents in spent solvent oil mixtures. Applicable analytes and their respective retention times are presented in Table 1.

1.2 The range of concentration determined by this method is 0.5 to 100% and is applicable only to concentrated organic matrices.

1.3 This method is based upon a gas chromatographic/flame ionization detection (GC/FID) procedure.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the samples are prepared for gas chromatography using the extraction procedure for waste solvent.

2.2 An aliquot of the extract is introduced into the gas chromatograph by direct injection. The solvents are separated by a capillary column and detected by a flame ionization detector.

3.0 INTERFERENCES

3.1 Potential interferences from the waste mixture are effectively reduced or removed by the vacuum distillation extraction procedure.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - Hewlett-Packard HP 5890 with Model 7673A autosampler and Model 3396 integrating recorder.

4.2 Column - Hewlett-Packard Ultra 2 (cross linked 5% phenyl methyl silicone gum) 50 meter x 0.32 mm ID with 0.52 µm film thickness

4.3 Detector - Flame Ionization Detector

4.4 Volumetric flasks - 10 ml Class A

4.5 Precision Balance - Capable of weighing 0.05 $\pm$ 0.01g

4.6 Pastuer Pipettes - 5 3/4" glass disposable

5.0 REAGENTS

5.1 Solvents - Chlorobenzene (ACS grade minimum with no interfering impurities)

5.2 Calibration Standards

Due to the high concentration range of this method, calibration standards may be prepared directly from the neat solvents.

5.2.1 Halogenated Solvents

ACS grade (99%+) solvents with no interfering peaks:

1,1,1-Trichloroethane
Trichloroethylene (trichloroethene)
Perchloroethylene (tetrachloroethene)
1,1,2-Trichloro-1,2,2-trifluoroethane (freon 113)
Methylene chloride

5.2.2 Non-Halogenated Solvents

ACS grade (99%+) solvents with no interfering peaks:

Methanol	n-Butyl alcohol
Ethanol	1,4-Dioxane
Acetone	Methylisobutyl ketone
2-Propanol	Toluene
t-Butyl alcohol	Methyl ethyl ketone

Nitromethane	Cyclohexene oxide
Butylene oxide	o-Xylene
sec-Butyl alcohol	m-Xylene
dioxolane	p-Xylene

5.2.3 Preparation of Standards

Calibration standards of the solvents are prepared at three concentration levels by weighing the appropriate mass of solvent in a tared 10 m*l* volumetric flask and diluting to the mark with chlorobenzene. Mixed standards may be prepared for the dilute standards. The procedure is:

- a clean, dry 10 m*l* volumetric flask is filled approximately half full with chlorobenzene. Allow the flask to stand approximately 10 minutes or until the wetted surfaces have dryed. Weigh the flask to the nearest 0.001 g.

- Using a Pastuer pipette, transfer the appropriate mass of solvent to the flask ensuring that the drops fall directly into the chlorobenzene without contacting the neck of the flask.

- Reweigh, dilute to the mark, stopper and mix by inverting the flask at least six times.

- Calculate the concentration in weight/volume percent from the net gain in weight. Correct for compound purity if purity is less than 98%.

- Repeat procedures for remainder of standards. For less concentrated standards, multiple compounds may be added.

- Transfer standards to the appropriate vials and cap with polyethylene lined closures.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sampling is completed by personnel at each Solvents Division facility according to protocols described in Section C of each facilities Part B Permit Application. Sample handling and preservation are outlined in Section 5.0 of the QAPP.

7.0 PROCEDURE

7.1 Extraction

The procedure for sample extraction presented in the Standard Operating Procedures.

7.2 Gas Chromatography Conditions

Initial oven temperature and holding time	35° - 6 minutes
Oven temperature program 1	4°C/minute
Second oven temperature and holding time	90°C - 0 minutes
Oven temperature program 2	10°C/minutes
Final oven temperature and holding time	180°C - 1 minute

Injector temperature	200°C
Detector temperature	250°C
Injector: Split-split ratio	1:100
Sample volume	1.0 µl
Carrier gas	Helium at 180 psi
Detector gas	Hydrogen at 18 psi
	Air at 45 psi

7.3 Calibration

7.3.1 External Standard Calibration Procedure

7.3.1.1 For each analyte of interest, prepare calibration standards at a minimum of three concentration levels by the procedure described in section 5.2.3.

7.3.1.2 Inject each calibration standard using the autosampler. Tabulate peak area response against the amount injected for

each compound of the standard. This ratio of area to amount injected is termed the calibration factor and is defined as:

$$\text{Calibration factor} = \frac{\text{Total Area of Peak}}{\%\ \text{component}}$$

If the percent relative standard deviation (%RSD) of the calibration factors is less than 20% over the working range, linearity throughout the origin is assumed and the average calibration factor may be used. If the %RSD is greater than 20%, then a calibration curve is calculated using linear regression analysis. The minimum acceptable correlation coefficient is 0.975.

7.3.1.3 The working calibration curve or calibration factor must be verified each working day by the inspection of one or more calibration standards. If the response for any analyte varies from the predicted response by more than ± 15%, a new calibration curve must be established for that analyte.

$$\%\ \text{difference} = \left(\frac{C_1 - C_2}{C_1}\right) \times 100$$

Where:

C_1 = Expected concentration

C_2 = Determined concentration

7.4 Gas Chromatographic Analysis

7.4.1 Inject 1.0 μl of the sample extract using the autosampler. If the response exceeds the linear range of the system, dilute the extract and reanalyze.

7.4.2 Calibrate the system (or confirm calibration) prior to conducting any analyses. A midlevel standard must also be injected at specific frequencies and at the end of the analytical batch. The determined concentration must be within ± 15% difference as compared to the expected concentration. If this criteria is exceeded, inspect the GC system to determine the cause and perform the necessary maintenance to correct the problem before recalibrating the system. All samples that were analyzed after the standard that exceeded the criteria are out of control and must be reanalyzed after the corrective action is taken.

7.4.3 Establish daily retention times for each analyte. Use absolute retention times for each analyte from the daily check standard as the midpoint for that day.

7.4.3.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time. No confirmation is necessary for the major degreasing solvents that are routinely determined. Compounds that are less frequently encountered will require an additional analytical technique for identification, if required. Tentative

identification is done more positively by spiking the sample with the suspected constituent and noting an increase in peak height. Sample would be sent to Detrex's RTI Lab in Detroit for positive ID, if necessary.

7.4.3.2 Validation of GC System Quantitative Performance

The midpoint calibration standard is used to evaluate this criterion. If any of the standards are outside of the daily retention time window, the system is out of control and corrective action will be necessary.

7.4.4 Sequence of Analysis

After the system has been calibrated and daily retention time calculated, the following sequence of analysis takes place:

1) Blank
2) 30 samples
3) Matrix Spike
4) Blank Spike
5) Duplicate
6) Calibration Check Standard(s)
7) Blank
8) 30 Samples

7.5 System Maintenance

These corrective actions may be required to remedy problems associated with poor response, shifting retention times, ghost peaks and unstable baselines.

7.5.1 System Bake-Out - To remedy problems associated with rising baselines and ghosts peaks, the chromatography system is baked out. This entails raising the oven temperature to the column conditioning temperature for two hours.

7.5.2 Septum Change - Unstable retention times and poor response may be attributed to a leaking injection port septum. Change septum per the maintenance manual guidelines.

7.5.3 Capillary Column maintenance - Ghost peaks and reduced response may be caused by dirty injection port inserts and front-end column contamination. Replace column insert with a clean-deactivated insert. Also, clipping the first few inches to a foot of the column may be necessary to remedy the problem.

7.5.4 Detector Maintenance-Reduced response may be caused by a dirty detector. Cool detector and clean as recommended in the maintenance manual. Recalibration of the system may be required after cleaning the detector.

7.6 Calculations

The concentration of each analyte in the sample is determined by calculating the peak response using the calibration factor or calibration curve determined in Step 7.3.1. The concentration of a specific analyte is calculated as follows:

$$\text{Concentration (\% wt/vol)} = \left(\frac{Ax}{cf} \right) \times D$$

where: Ax = Response for analyte in sample area units

cf = Calibration Factor - area units per unit concentrations

D = Dilution Factor (if any)

8.0 QUALITY CONTROL

8.1 Reagent Blanks - To demonstrate that the analytical system is not introducing analytes or interferences, reagent blanks consisting of chlorobenzene are processed with the samples. The blanks are carried completely through the extraction and measurement steps and are analyzed at a frequency of 1% of all samples or one per day; whichever is more frequent.

8.2 Accuracy Control

8.2.1 Matrix Spikes Samples - to demonstrate that the sample matrix is not affecting the accuracy of the measurement, matrix spike samples are analyzed. These samples consist of a sample aliquot that has been fortified with a known amount of analyte. The results are expressed as percent recovery (%R) of added analyte and are calculated by:

$$\%R = \left(\frac{SSR - SR}{SA} \right) \times 100$$

where SSR = Spike Sample result

SR = Sample result

SA = Spike added

Matrix spike samples are processed and analyzed at a frequency of one per week. Due to the nature of the samples, only a specific sub set of analytes will be spiked into the sample.

8.2.2 Blank Spike Samples - To demonstrate the efficiency of the extraction and analytical procedures, blank spike samples are analyzed. These samples consist of an artificial matrix that is spiked with a known amount of all analytes of interest. The results are expressed as percent recovery (%R) of added analyte and are calculated by

$$\%R = \left(\frac{BSR - BR}{SA} \right) \times 100$$

where BSR = Blank Spike Result

BR = Blank result

SA = Spike added

Blank spike samples are processed and analyzed at a frequency of one per week.

8.3 Precision Control

To demonstrate the repeatability of the extraction and analytical procedures, duplicate sample analyses are performed. Due to the fact that there will be detectable analytes in every sample, this type of precision control is preferable to matrix spike, matrix spike duplicate analysis. Equal aliquots of a sample are processed and analyzed. The results are expressed as relative percent difference and are calculated by:

$$RPD = \frac{|SR_1 - SR_2|}{\left(\frac{SR_1 + SR_2}{2}\right)} \times 100$$

Duplicate samples are analyzed at a frequency of 1% of all samples or one per day, whichever is more frequent. RPD is calculated for %NVR and for detectable analytes.

where: SR_1 = sample result 1

SR_2 = sample result 2

8.4 Control Limits and Corrective Actions

8.4.1 Reagent Blanks

Reagent blanks should be free from interfering peaks and analytes of interest. Should there be evidence of contamination, the following should be investigated to locate and eliminate the source.

-Purity of solvent used for blank
-Cleanliness of glassware
-Use of lubricating grease on distillation apparatus
-Adequate rinsing of GC syringe
-Carryover from previous sample analysis
-Contaminated GC system

If the problem is determined to be isolated only to the reagent blank (in the case of carryover) no corrective action is required. Should the problem be determined to be systematic, re-extraction and analysis of the samples associated with the reagent blank must take place. Documentation of corrective actions taken is recorded in a bound laboratory notebook.

8.4.2 Matrix Spike Samples

Recovery limits for spike compounds are listed below:

1,1,1-Trichloroethane	75-125%
Trichloroethylene	75-125%
Perchloroethylene	75-125%
Freon	65-125%
Methylene chloride	75-125%

Should recovery limits be outside the acceptance criteria, the following should be investigated or conducted:

-Re-analysis of the matrix spike sample
-Evaluation of results of sample analysis. Concentrated samples may cause the spiked sample to be outside the linear range of the instrument. Dilute and re-analyze.
-Sample homogeneity
-Loss of instrument calibration - analyze a check standard for calibration verification

Should re-analysis of the matrix spike sample still indicate an out-of-control situation, a new sample will be chosen for matrix spike analysis. If analysis still yields out-of-control results, a thorough investigation must be conducted and the finding documentated. All samples processed from the last in-control point must be reprocessed and re-analyzed.

Should results of matrix spike sample analysis indicate an isolated problem associated with the blank spike sample, re-extraction and re-analysis is not required. In all other cases, re-extraction and re-analysis from the last in-control sample must occur.

8.4.3 Blank Spike Samples - Recovery limits for blank spike compounds are listed below:

1,1,1-Trichloroethane	75-125%
Trichloroethylene	75-125%
Perchloroethylene	75-125%
Freon	65-125%
Methylene chloride	75-125%

Should the above recovery limits be exceeded, the following should be investigated or conducted:

-Re-analysis of the blank spike sample
-Contamination of distillation due to loss of vacuum or inappropriate bath temperature
-Loss of calibration of GC. A check standard should be analyzed.

8.4.4 Replicate sample analysis - RPD limits for NVR and detected compounds are 30%. Should this criterion be violated, the following should be investigated or conducted.

-Re-analysis of samples
-Contamination of distillation and GC systems
-Homogeneity of sample matrix
-Inefficient distillations

Should re-analysis of samples not elicit in-control results, a new sample from the batch will be chosen for replicate analysis. If the results of the new replicates still indicate an out-of-control situation, a thorough investigation must be conducted and the findings documented. All samples processed from the last in-control point must be re-extracted and re-analyzed.

9.0 METHOD PERFORMANCE

At this time, the performance of the method is still being evaluated.

10.0 REFERENCES

1) Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, EPA SW-846 Methods 8000, 8010 and 8015.

2) 1989 Annual Book of ASTM Standards, ASTM Volume 15.05 Methods D 3742 and D-3447, Volume 14.01.

9. Quality Control

9.1 Quality control check samples and duplications should be performed at an action level specified by the laboratory and at an appropriate frequency.

9.2 Flammability standards should be maintained for analyst training and as reference guides. Examples are given in the following table.

Flammability Potential Screening Analysis

Methyl alcohol (methanol)	Difficult-to-see flame: flammable at or below room temperature.
Glacial acetic acid	Flammable at temperatures above normal room temperature.
Hendecane (n-undecane)	Flammable at temperatures higher than glacial acetic acid.
Kerosene (Fuel Oil No. 1)	Flammable over a range of temperatures higher than normal room temperature.
pXylene	Flammable at or above room temperature: freezes at 13°C. A spiked soil sample frozen at 0°C demonstrates interference.
Chlorofluorocarbon 113 (1,1,2-trichloro-1,2,2-trifluoroethane)	Warning: phosgene formation likely; see 13.4.3.1.

10. Precision and Bias

10.1 No statement is made about either the precision or bias of these test methods since the result merely states whether there is conformance to the criteria for success specified in the procedure.

TEST METHOD A—EXPOSURE TO HEAT AND FLAME

11. Interferences

11.1 Drafts in the laboratory fume hood where the test is performed could cause excessive cooling and false negative results. A properly operating fume hood with a face velocity of 150 ft/min should provide consistent, usable results.

11.2 Ignition sources that provide excessive heating rates alone or combined with a very small test portion may obscure results. That is, the sample may be decomposed, sintered, fused, evaporated, or otherwise consumed before positive or negative evidence of flammability is observed. The use of sufficiently large test portions and of heating rates typical of a bunsen burner should resolve this problem.

11.3 An inadequate source of heating could limit the volatilization of flammable components and provide false negative results.

11.4 Difficult-to-observe flames resulting from the burning of certain compounds (for example, methanol) could provide false negative results. If the presence of these compounds is suspected, the presence of flammability might be confirmed by the insertion of a watchglass just above the test material. The watchglass is then examined for products of combustion (for example, moisture and soot).

NOTE 1—The watchglass method cannot distinguish between vaporized water or water produced by combustion.

11.5 An improperly adjusted gas burner (for example, with an insufficiently aerated flame) could introduce raw gas into or immediately above the sample. This raw gas could briefly support a flame after the source of ignition is removed, providing the appearance of a flammable sample and a false positive result.

12. Apparatus

12.1 *Gas Burner*, (for example, a bunsen burner) with an adjustable air shutter and an adjustable gas orifice is needed. The gas burner and fuel supply line must be appropriate to the gas supplied: natural gas, artificial gas (including propane and butane), or liquified petroleum gas (LP gas or LPG). Where a gas supply line cannot be provided, a propane torch may be substituted.

12.2 *Lighter*, (for example, piezo lighter) for burner is required.

12.3 *Aluminum Weighing Boats* or other non-flammable containers are needed.

12.3.1 Caution—Weighing boats of material other than aluminum should be used if the testing materials react with aluminum, for example, caustics.

12.4 *Watchglass*.

12.5 *Large Beaker, Tongs, Asbestos-Free High-Temperature Gloves or Mittens*, or other apparatus as needed to extinguish burning materials.

13. Procedure

13.1 Light a gas burner and adjust to a typically blue flame that is not readily blown out. A yellow flame easily affected by drafts indicates insufficient air (the air:fuel ratio is too low). A sharp, blue flame is good. (If the flame rises above the burner head, is very difficult to light, or tends to extinguish itself, indicates that too much air or too much air and fuel are being supplied to the burner.)

13.2 Place a sufficient amount (approximately 5 g) of a test sample in an aluminum weighing boat or other nonflammable container.

13.3 Using a gas burner, hold the flame immediately above the test sample for 2 to 3 s without touching the visible flame to the sample.

13.3.1 If ignition (a flash or burning) is observed before or after the source of ignition (the flame of the burner) is removed, the sample is said to have a positive flammability potential. A positive result may require further investigation (see 13.4.1).

13.3.2 The confirmation of flammability may require the use of a watchglass (see 11.4).

13.3.3 If there is no ignition, proceed to 13.4.

13.4 Using a gas burner, briefly (for at least 15 s) apply the flame to the sample in an attempt to ignite the sample.

13.4.1 If the sample ignites, the sample is said to have a positive flammability potential.

13.4.1.1 When more accurate waste characterization is necessary, liquid samples may be quantified using a closed-cup flash point tester.

13.4.1.2 Solids with a positive flammability potential should be further investigated.

13.4.2 If the sample decomposes, boils (if a liquid), or otherwise fails to ignite after at least 15 s of continuous sample heating by the burner flame, the flammability potential is reported as negative.

13.4.3 Halogenated solvents typically give off visible vapors that may result in a false positive flammability potential.

NOTE 2: Warning—Phosgene, an extremely toxic gas, is a combustion product of halogenated compounds burned in air.

13.5 Shut off the gas burner when not in use. Extinguish a

burning sample by setting an aluminum weighing boat or watchglass atop the one containing the burning sample, (or invert a spoutless beaker over the sample container and all). Use of tongs or high temperature gloves or mittens may be necessary to handle the equipment.

TEST METHOD B—EXPOSURE TO SPARK SOURCE

14. Apparatus

14.1 *Oven Gloves.*

14.2 *Flint Lighter*, the type typically used to light an air/acetylene torch is required.

14.3 *Disposable 250-mL Beaker*, of plastic is required.

14.4 *Watchglass*, 100 mm.

14.5 *Metal Vessel* (with lid), of adequate depth and diameter to contain beaker and watchglass is needed.

14.6 *Thermometer.*

15. Procedure

15.1 Place approximately 100 mL of the representative sample of the material to be tested into the plastic beaker (see 7.3).

15.2 Place the plastic beaker in the steel vessel, cover the beaker with the watchglass, and allow to stand at ambient conditions for 5 min.

15.3 Record the ambient temperature.

15.4 Remove the watchglass, place the igniter immediately above the waste and strike it several times to produce sparks.

15.5 If the material does catch fire and burn, extinguish the flames by immediately placing the lid on the steel vessel, thus smothering the fire and report as positive flammability potential.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

STANDARD OPERATING PROCEDURES FOR THE EXTRACTION OF SPENT SOLVENTS

STANDARD OPERATING PROCEDURES FOR THE EXTRACTION OF SPENT SOLVENTS

1.0 SCOPE

This method describes a sampling preparation procedure for the determination of halogenated solvents in a spent halogenated solvent oil mixture.

2.0 SUMMARY OF METHOD

A sample of spent halogenated solvent waste is weighed in a tared, flat bottom flask. The flask is connected to a rotary vacuum evaporator and the solvent is vacuum distilled from the mixture, the weight of residue is determined, and the total non-volatile percentage calculated.

3.0 APPARATUS AND MATERIALS

3.1 Rotary vacuum evaporator with heating bath.

3.2 250 m*l* flat bottom flasks:

3.3 Precision Balance: capable of measuring to .005 ± 001 g.

3.4 Vacuum Apparatus: water aspirator.

4.0 PROCEDURE

Preheat water bath to 90°C. Pour approximately 30 m*l* of sample into a tared flat bottom flask, weigh and record initial weight. Attach flask to rotary vacuum evaporator, apply vacuum, lower flask into water bath and roto-evaporate solvent from solvent-oil sample until condensate ceases flowing from the condensor. Remove flask from evaporator, dry, reweigh and record. Remove (pour) distillate from receiving flask into clean sample container and store for analysis.

5.0 DETERMINATION OF % NON-VOLATILE RESIDUE

The calculation of % non-volatile residue is as follows:

$$\frac{R}{S} \times 100 = \% \text{ NV}$$

R=Mass of residue after roto-evaporation

S=Mass of waste solvent before roto-evaporation

6.0 The distillate is subsequently analyzed for individual halogenated solvents by gas chromatography.

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENTS IN SPENT SOLVENTS BY GAS CHROMATOGRAPHY

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENTS IN SPENT SOLVENTS BY GAS CHROMATOGRAPHY

1.0 SCOPE

1.1 This method is used for the determination of recoverable halogenated and non-halogenated solvents in spent solvent oil mixtures. Applicable analytes and their respective retention times are presented in Table 1.

1.2 The range of concentration determined by this method is 0.5 to 100% and is applicable only to concentrated organic matrices.

1.3 This method is based upon a gas chromatographic/flame ionization detection (GC/FID) procedure.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the samples are prepared for gas chromatography using the extraction procedure for waste solvent.

2.2 An aliquot of the extract is introduced into the gas chromatograph by direct injection. The solvents are separated by a capillary column and detected by a flame ionization detector.

TABLE 1
RETENTION TIMES FOR ORGANIC SOLVENTS

Compound	*Retention Time (minutes)*[1]
Methanol	2.83
Ethanol	3.16
Acetone	3.46
2-Propanol	3.51
t-Butyl alcohol	3.77
Freon	3.91
Methylene chloride	3.99
Nitromethane	4.45
Butylene oxide	5.19
Methylethylketone	5.22
sec-Butyl alcohol	5.32
Dioxolane	5.63
1,1,1-Trichloroethane	6.76
n-Butyl alcohol	7.69
Trichloroethylene	9.01
1,4-Dioxane	9.43
Methylisobutyl ketone	11.0
Toluene	12.4
Perchloroethylene	14.8
Cyclohexene oxide	17.8
o-Xylene	17.6
m-Xylene	18.2
p-Xylene	19.2

[1]Retention times are approximate and for illustration purposes only. Daily operating conditions will affect actual retention times.

3.0 INTERFERENCES

3.1 Potential interferences from the waste mixture are effectively reduced or removed by the vacuum distillation extraction procedure.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - Hewlett-Packard HP 5890 with Model 7673A autosampler and Model 3396 integrating recorder.

4.2 Column - Hewlett-Packard Ultra 2 (cross linked 5% phenyl methyl silicone gum) 50 meter x 0.32 mm ID with 0.52 µm film thickness

4.3 Detector - Flame Ionization Detector

4.4 Volumetric flasks - 10 ml Class A

4.5 Precision Balance - Capable of weighing 0.05 ± 0.01g

4.6 Pastuer Pipettes - 5 3/4" glass disposable

5.0 REAGENTS

5.1 Solvents - Chlorobenzene (ACS grade minimum with no interfering impurities)

5.2 Calibration Standards

Due to the high concentration range of this method, calibration standards may be prepared directly from the neat solvents.

5.2.1 Halogenated Solvents

ACS grade (99%+) solvents with no interfering peaks:

1,1,1-Trichloroethane
Trichloroethylene (trichloroethene)
Perchloroethylene (tetrachloroethene)
1,1,2-Trichloro-1,2,2-trifluoroethane (freon 113)
Methylene chloride

5.2.2 Non-Halogenated Solvents

ACS grade (99%+) solvents with no interfering peaks:

Methanol	n-Butyl alcohol
Ethanol	1,4-Dioxane
Acetone	Methylisobutyl ketone
2-Propanol	Toluene
t-Butyl alcohol	Methyl ethyl ketone

Nitromethane	Cyclohexene oxide
Butylene oxide	o-Xylene
sec-Butyl alcohol	m-Xylene
dioxolane	p-Xylene

5.2.3 Preparation of Standards

Calibration standards of the solvents are prepared at three concentration levels by weighing the appropriate mass of solvent in a tared 10 m*l* volumetric flask and diluting to the mark with chlorobenzene. Mixed standards may be prepared for the dilute standards. The procedure is:

- a clean, dry 10 m*l* volumetric flask is filled approximately half full with chlorobenzene. Allow the flask to stand approximately 10 minutes or until the wetted surfaces have dryed. Weigh the flask to the nearest 0.001 g.

- Using a Pastuer pipette, transfer the appropriate mass of solvent to the flask ensuring that the drops fall directly into the chlorobenzene without contacting the neck of the flask.

- Reweigh, dilute to the mark, stopper and mix by inverting the flask at least six times.

- Calculate the concentration in weight/volume percent from the net gain in weight. Correct for compound purity if purity is less than 98%.

- Repeat procedures for remainder of standards. For less concentrated standards, multiple compounds may be added.

- Transfer standards to the appropriate vials and cap with polyethylene lined closures.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sampling is completed by personnel at each Solvents Division facility according to protocols described in Section C of each facilities Part B Permit Application. Sample handling and preservation are outlined in Section 5.0 of the QAPP.

7.0 PROCEDURE

7.1 Extraction

The procedure for sample extraction presented in the Standard Operating Procedures.

7.2 Gas Chromatography Conditions

Initial oven temperature and holding time	35° - 6 minutes
Oven temperature program 1	4°C/minute
Second oven temperature and holding time	90°C - 0 minutes
Oven temperature program 2	10°C/minutes
Final oven temperature and holding time	180°C - 1 minute

Injector temperature	200°C
Detector temperature	250°C
Injector: Split-split ratio	1:100
Sample volume	1.0 µl
Carrier gas	Helium at 180 psi
Detector gas	Hydrogen at 18 psi
	Air at 45 psi

7.3 Calibration

7.3.1 External Standard Calibration Procedure

7.3.1.1 For each analyte of interest, prepare calibration standards at a minimum of three concentration levels by the procedure described in section 5.2.3.

7.3.1.2 Inject each calibration standard using the autosampler. Tabulate peak area response against the amount injected for

each compound of the standard. This ratio of area to amount injected is termed the calibration factor and is defined as:

$$\text{Calibration factor} = \frac{\text{Total Area of Peak}}{\%\ \text{component}}$$

If the percent relative standard deviation (%RSD) of the calibration factors is less than 20% over the working range, linearity throughout the origin is assumed and the average calibration factor may be used. If the %RSD is greater than 20%, then a calibration curve is calculated using linear regression analysis. The minimum acceptable correlation coefficient is 0.975.

7.3.1.3 The working calibration curve or calibration factor must be verified each working day by the inspection of one or more calibration standards. If the response for any analyte varies from the predicted response by more than ± 15%, a new calibration curve must be established for that analyte.

$$\%\ \text{difference} = \left(\frac{C_1 - C_2}{C_1}\right) \times 100$$

Where:

C_1 = Expected concentration

C_2 = Determined concentration

7.4 Gas Chromatographic Analysis

7.4.1 Inject 1.0 µl of the sample extract using the autosampler. If the response exceeds the linear range of the system, dilute the extract and reanalyze.

7.4.2 Calibrate the system (or confirm calibration) prior to conducting any analyses. A midlevel standard must also be injected at specific frequencies and at the end of the analytical batch. The determined concentration must be within ± 15% difference as compared to the expected concentration. If this criteria is exceeded, inspect the GC system to determine the cause and perform the necessary maintenance to correct the problem before recalibrating the system. All samples that were analyzed after the standard that exceeded the criteria are out of control and must be reanalyzed after the corrective action is taken.

7.4.3 Establish daily retention times for each analyte. Use absolute retention times for each analyte from the daily check standard as the midpoint for that day.

7.4.3.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time. No confirmation is necessary for the major degreasing solvents that are routinely determined. Compounds that are less frequently encountered will require an additional analytical technique for identification, if required. Tentative

identification is done more positively by spiking the sample with the suspected constituent and noting an increase in peak height. Sample would be sent to Detrex's RTI Lab in Detroit for positive ID, if necessary.

7.4.3.2 Validation of GC System Quantitative Performance

The midpoint calibration standard is used to evaluate this criterion. If any of the standards are outside of the daily retention time window, the system is out of control and corrective action will be necessary.

7.4.4 Sequence of Analysis

After the system has been calibrated and daily retention time calculated, the following sequence of analysis takes place:

1) Blank
2) 30 samples
3) Matrix Spike
4) Blank Spike
5) Duplicate
6) Calibration Check Standard(s)
7) Blank
8) 30 Samples

7.5 System Maintenance

These corrective actions may be required to remedy problems associated with poor response, shifting retention times, ghost peaks and unstable baselines.

7.5.1 System Bake-Out - To remedy problems associated with rising baselines and ghosts peaks, the chromatography system is baked out. This entails raising the oven temperature to the column conditioning temperature for two hours.

7.5.2 Septum Change - Unstable retention times and poor response may be attributed to a leaking injection port septum. Change septum per the maintenance manual guidelines.

7.5.3 Capillary Column maintenance - Ghost peaks and reduced response may be caused by dirty injection port inserts and front-end column contamination. Replace column insert with a clean-deactivated insert. Also, clipping the first few inches to a foot of the column may be necessary to remedy the problem.

7.5.4 Detector Maintenance-Reduced response may be caused by a dirty detector. Cool detector and clean as recommended in the maintenance manual. Recalibration of the system may be required after cleaning the detector.

7.6 Calculations

The concentration of each analyte in the sample is determined by calculating the peak response using the calibration factor or calibration curve determined in Step 7.3.1. The concentration of a specific analyte is calculated as follows:

$$\text{Concentration (\% wt/vol)} = \left(\frac{Ax}{cf} \right) \times D$$

where: Ax = Response for analyte in sample area units

cf = Calibration Factor - area units per unit concentrations

D = Dilution Factor (if any)

8.0 QUALITY CONTROL

8.1 Reagent Blanks - To demonstrate that the analytical system is not introducing analytes or interferences, reagent blanks consisting of chlorobenzene are processed with the samples. The blanks are carried completely through the extraction and measurement steps and are analyzed at a frequency of 1% of all samples or one per day; whichever is more frequent.

8.2 Accuracy Control

8.2.1 Matrix Spikes Samples - to demonstrate that the sample matrix is not affecting the accuracy of the measurement, matrix spike samples are analyzed. These samples consist of a sample aliquot that has been fortified with a known amount of analyte. The results are expressed as percent recovery (%R) of added analyte and are calculated by:

$$\%R = \left(\frac{SSR - SR}{SA} \right) \times 100$$

where SSR = Spike Sample result

SR = Sample result

SA = Spike added

Matrix spike samples are processed and analyzed at a frequency of one per week, whichever is more frequent. Due to the nature of the samples, only a specific sub set of analytes will be spiked into the sample.

8.2.2 Blank Spike Samples - To demonstrate the efficiency of the extraction and analytical procedures, blank spike samples are analyzed. These samples consist of an artificial matrix that is spiked with a known amount of all analytes of interest. The results are expressed as percent recovery (%R) of added analyte and are calculated by

$$\%R = \left(\frac{BSR - BR}{SA} \right) \times 100$$

where BSR = Blank Spike Result

BR = Blank result

SA = Spike added

Blank spike samples are processed and analyzed at a frequency of one per week.

8.3 Precision Control

To demonstrate the repeatability of the extraction and analytical procedures, duplicate sample analyses are performed. Due to the fact that there will be detectable analytes in every sample, this type of precision control is preferable to matrix spike, matrix spike duplicate analysis. Equal aliquots of a sample are processed and analyzed. The results are expressed as relative percent difference and are calculated by:

$$RPD = \frac{|SR_1 - SR_2|}{\left(\frac{SR_1 + SR_2}{2}\right)} \times 100$$

Duplicate samples are analyzed at a frequency of 1% of all samples or one per day, whichever is more frequent. RPD is calculated for %NVR and for detectable analytes.

where: SR_1 = sample result 1

SR_2 = sample result 2

8.4 Control Limits and Corrective Actions

8.4.1 Reagent Blanks

Reagent blanks should be free from interfering peaks and analytes of interest. Should there be evidence of contamination, the following should be investigated to locate and eliminate the source.

-Purity of solvent used for blank
-Cleanliness of glassware
-Use of lubricating grease on distillation apparatus
-Adequate rinsing of GC syringe
-Carryover from previous sample analysis
-Contaminated GC system

If the problem is determined to be isolated only to the reagent blank (in the case of carryover) no corrective action is required. Should the problem be determined to be systematic, re-extraction and analysis of the samples associated with the reagent blank must take place. Documentation of corrective actions taken is recorded in a bound laboratory notebook.

8.4.2 Matrix Spike Samples

Recovery limits for spike compounds are listed below:

1,1,1-Trichloroethane	75-125%
Trichloroethylene	75-125%
Perchloroethylene	75-125%
Freon	65-125%
Methylene chloride	75-125%

Should recovery limits be outside the acceptance criteria, the following should be investigated or conducted:

-Re-analysis of the matrix spike sample
-Evaluation of results of sample analysis. Concentrated samples may cause the spiked sample to be outside the linear range of the instrument. Dilute and re-analyze.
-Sample homogeneity
-Loss of instrument calibration - analyze a check standard for calibration verification

Should re-analysis of the matrix spike sample still indicate an out-of-control situation, a new sample will be chosen for matrix spike analysis. If analysis still yields out-of-control results, a thorough investigation must be conducted and the finding documentated. All samples processed from the last in-control point must be reprocessed and re-analyzed.

Should results of matrix spike sample analysis indicate an isolated problem associated with the blank spike sample, re-extraction and re-analysis is not required. In all other cases, re-extraction and re-analysis from the last in-control sample must occur.

8.4.3 Blank Spike Samples - Recovery limits for blank spike compounds are listed below:

1,1,1-Trichloroethane	75-125%
Trichloroethylene	75-125%
Perchloroethylene	75-125%
Freon	65-125%
Methylene chloride	75-125%

Should the above recovery limits be exceeded, the following should be investigated or conducted:

-Re-analysis of the blank spike sample
-Contamination of distillation due to loss of vacuum or inappropriate bath temperature
-Loss of calibration of GC. A check standard should be analyzed.

8.4.4 Replicate sample analysis - RPD limits for NVR and detected compounds are 30%. Should this criterion be violated, the following should be investigated or conducted.

-Re-analysis of samples
-Contamination of distillation and GC systems
-Homogeneity of sample matrix
-Inefficient distillations

Should re-analysis of samples not elicit in-control results, a new sample from the batch will be chosen for replicate analysis. If the results of the new replicates still indicate an out-of-control situation, a thorough investigation must be conducted and the findings documented. All samples processed from the last in-control point must be re-extracted and re-analyzed.

9.0 METHOD PERFORMANCE

At this time, the performance of the method is still being evaluated.

10.0 REFERENCES

1) Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, EPA SW-846 Methods 8000, 8010 and 8015.

2) 1989 Annual Book of ASTM Standards, ASTM Volume 15.05 Methods D 3742 and D-3447, Volume 14.01.

ATTACHMENT C-4

SAMPLE LABORATORY REPORTS

CONESTOGA-ROVERS & ASSOCIATES

MEMORANDUM

TO: [redacted]

FROM: Steve Day

DATE: August 2, 1991

REF. NO: 2471

RE: Waste Compatibility for Materials Stored at the Detrex Corporation Easton Avenue Detroit, Michigan Facility

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In reference to Item C-6 of the draft Notice of Deficiency (NOD) issued by the Michigan Department of Natural Resources (MDNR) for the above-reference facility, attached is a detailed review of the compatibility analysis performed for the wastes stored at the Detrex Corporation (Detrex) Facility in Melrose Park, Illinois. The wastes stored at the Melrose Park facility are essentially the same type of waste as the Eaton Avenue facility and, consequently, the waste compatibility analysis applies. The compatibility analysis was performed as described in "A Method for Determining the Compatibility of Hazardous Wastes" EPA-600/2-80-076, April 1980.

It should be noted that the composite sampling of the ten drums of waste type per generator will also act as a field compatibility test. Should incompatible materials be present in the drums, this would be discovered during the composite sampling. In addition, Detrex's familiarity with the waste and historical record of no reported incidences involving waste capability should preclude the need to perform additional compatibility testing per waste type.

SCD/lo/2

CONESTOGA-ROVERS & ASSOCIATES
MEMORANDUM

TO: Bruce McConnell

FROM: [redacted]

DATE: March 1, 1991

REF. NO.: 2471

RE: Waste Compatibility for Materials Stored at the Detrex Corporation Facility in Melrose Park, Illinois

---

In reference to the hazardous waste stored at the Detrex Corporation facility located in Melrose Park, Illinois, the following text and tables provide information regarding the compatibility of the stored wastes. The following evaluation was based on information supplied by Detrex and was conducted on all possible compounds that might be found in the waste streams. Evaluation of the compatibility of the wastes was conducted with the use of a USEPA document[1] that provides methodology for determining waste compatibility.

The method consists of a step by step compatibility analysis procedure and the use of a compatibility chart. Wastes to be mixed or combined are identified and classified, and the chart is used to predict the compatibility of the wastes on mixing. Worksheets, which were provided with the document, were used to establish a matrix of the different combinations of compounds and list any hazardous reactions. Binary combinations that produce hazardous reactions are summarized in Attachment 1. Individual worksheets are provided as Attachment 2.

Elemental metals and reactivity group 17 solvents cause the generation of heat and fire when mixed as illustrated on Worksheet 1. It is unlikely that these metals would be found in significant amounts to cause these reactions. In addition, any metal-solvent reactions would have previously taken place since the metals would already be in contact with group 17 solvents. Barium is the most reactive of the listed metals as illustrated on worksheets 1, 2, 3 and 4, but only in the elemental state. It is unlikely that significant quantities of elemental barium will be found at the Melrose Park facility.

Solvents with reactivity group numbers 16, 17, 19 and 27 found at the facility are compatible with each other as illustrated on worksheets 5, 6, 7 and 8. Pyridine when mixed with metals or group 17 solvents cause hazardous reactions. Pyridine is not used as a degreasing agent or stabilizer for the degreasing solvent and is not expected to be present in the waste at significant concentrations. Vinyl chloride when mixed with metals also causes hazardous reactions as can be seen from worksheets 4, 7, and

---

[1] "A Method for Determining the Compatibility of Hazardous Wastes", EPA-600/2-80-076, April 1980

8. Vinyl chloride (monomer) exists as a gas in its reactive form and is not expected to be present in the waste at significant concentrations. As previously discussed, any reactions involving pyridine and vinyl chloride would occur prior to Detrex receiving the waste.

BC/ko/1

ATTACHMENT 1

A SUMMARY OF HAZARDOUS REACTIONS THAT OCCUR WITH BINARY COMBINATIONS FOR WASTE STORED AT THE DETREX FACILITY IN MELROSE PARK, ILLINOIS

Binary Combinations	*Reaction*[1]
Barium - All compounds	extremely reactive
Cadmium - 1,1,1-Trichloroethane	H, F
Cadmium - Trichloroethylene	H, F
Cadmium - Perchloroethlyene	H, F
Cadmium - Methylene Chloride	H, F
Cadmium - Trichlorotrifluoroethane (Freon)	H, F
Chromium - 1,1,1-Trichloroethane	H, F
Chromium - Trichloroethylene	H, F
Chromium - Perchloroethylene	H, F
Chromium - Methylene Chloride	H, F
Chromium - Trichlorotrifluoroethane	H, F
Lead - 1,1,1-Trichloroethane	H, F
Lead - Trichloroethylene	H, F
Lead - Perchloroehtlyene	H, F
Lead - Methylene Chloride	H, F
Lead - Trichlorotrifluoroethane	H, F
Selenium - 1,1,1-Trichloroethane	H, F, E
Selenium - Trichloroethylene	H, F, E
Selenium - Perchloroethylene	H, F, E
Selenium - Methylene Chloride	H, F, E
Selenium - Trichlorotrifluoroethane	H, F, E
Carbon Tetrachloride - Cadmium	H, F
Carbon Tetrachloride - Chromium	H, F
Carbon Tetrachloride - Lead	H, F
Carbon Tetrachloride - Selenium	H, F, E
Chlorobenzene - Cadmium	H, F
Chlorobenzene - Chromium	H, F
Chlorobenzene - Lead	H, F
Chlorobenzene - Selenium	H, F, E
Chloroform - Cadmium	H, F
Chloroform - Chromium	H, F
Chloroform - Lead	H, F
Chloroform - Selenium	H, F, E

ATTACHMENT 1

A SUMMARY OF HAZARDOUS REACTIONS THAT OCCUR WITH BINARY COMBINATIONS FOR WASTE STORED AT THE DETREX FACILITY IN MELROSE PARK, ILLINOIS

Binary Combinations	*Reaction*[1]
1,4-Dichlorobenzene - Cadmium	H, F
1,4-Dichlorobenzene - Chromium	H, F
1,4-Dichlorobenzene - Lead	H, F
1,4-Dichlorobenzene - Selenium	H, F, E
1,2-Dichloroethane - Cadmium	H, F
1,2-Dichloroethane - Chromium	H, F
1,2-Dichloroethane - Lead	H, F
1,2-Dichloroethane - Selenium	H, F, E
1,1-Dichloroethylene - Cadmium	H, F
1,1-Dichloroethylene - Chromium	H, F
1,1-Dichloroethylene - Lead	H, F
1,1-Dichloroethylene - Selenium	H, F, E
Vinyl Chloride - Arsenic	P, H
Vinyl Chloride - Cadmium	P, H
Vinyl Chloride - Chromium	P, H
Vinyl Chloride - Lead	P, H
Vinyl Chloride - Mercury	P, H
Vinyl Chloride - Selenium	P, H, E
Pyridine - Arsenic	P, H
Pyridine - Cadmium	P, H
Pyridine - Chromium	P, H
Pyridine - Lead	P, H
Pyridine - Mercury	P, H
Pyridine - Selenium	P, H
Pyridine - 1,1,1-Trichloroethane	H, GT
Pyridine - Trichloroethylene	H, GT
Pyridine - Perchloroethylene	H, GT
Pyridine - Methylene Chloride	H, GT
Pyridine - Trichlorotrifluoroethane	H, GT

ATTACHMENT 1

A SUMMARY OF HAZARDOUS REACTIONS THAT OCCUR WITH BINARY COMBINATIONS FOR WASTE STORED AT THE DETREX FACILITY IN MELROSE PARK, ILLINOIS

Binary Combinations	*Reaction*[1]
Pyridine - Carbon Tetrachloride	H, GT
Pyridine - Chlorobenzene	H, GT
Pyridine - Chloroform	H, GT
Pyridine - 1,4-Dichlorobenzene	H, GT
Pyridine - 1,2-Dichloroethane	H, GT
Pyridine - 1,1-Dichloroethylene	H, GT
Pyridine - Vinyl Chloride	H, GT

2471/KAO

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[1]H -Heat generation
GT -Toxic gas generation
F -Fire
P -Violent polymerization
S -Solubilization of toxic substances
E -Explosion

ATTACHMENT 2

WASTE EVALUATION WORKSHEETS

Waste A _____ Source _____

Waste B _____ Source _____

Name of Waste Evaluation _____ Date _____

WASTE A / WASTE B		Name	1,1,1-Trichloroethane	Trichlorethylene	Perchloroethylene	Methylene Chloride	Trichlorotrifluroethane			
Name	Reactivity Group No.	Reactivity Group No.	17	17	17	17	17			
Arsenic	24									
Barium	21,24,107		X	X	X	X	X			
Cadmium	23,24		H,F	H,F	H,F	H,F	H,F			
Chromium	23,24		H,F	H,F	H,F	H,F	H,F			
Lead	23,24		H,F	H,F	H,F	H,F	H,F			
Mercury	24									
Selenium	22,23,24		H,F,E	H,F,E	H,F,E	H,F,E	H,F,E			

Note: Refer to Figure 6 for the definitions of the Reaction Code entered on the squares of this worksheet.

Figure 2. Worksheet for determining hazardous waste compatibility.

H - Heat generation X - Extremely reactive

E - Explosion

F - Fire

Waste A _____ Source _____

Waste B _____ Source _____

Name of Waste Evaluation _____ Date _____

WASTE A / WASTE B		Arsenic 24	Barium 21,24,107	Cadmium 23,24	Chromium 23,24	Lead 23,24	Mercury 24	Selenium 22,23,24	
Name	Reactivity Group No.								
Arsenic	24		X						
Barium	21,24,107	X		X	X	X	X	X	
Cadmium	23,24		X						
Chromium	23,24		X						
Lead	23,24		X						
Mercury	24		X						
Selenium	22,23,24		X						

Note: Refer to Figure 6 for the definitions of the Reaction Code entered on the squares of this worksheet.

Figure 2. Worksheet for determining hazardous waste compatibility.

X - Extremely reactive

Waste A _____ Source _____

Waste B _____ Source _____

Name of Waste Evaluation _____ Date _____

WASTE A / WASTE B — Name	Reactivity Group No.	Arsenic 24	Barium 21,24,107	Cadmium 23,24	Chromium 23,24	Lead 23,24	Mercury 24	Selenium 22,23,24
Benzene	16		X					
Carbon Tetrachloride	17		X	H,F	H,F	H,F		H,F,E
Chlorobenzene	17		X	H,F	H,F	H,F		H,F,E
Chloroform	17		X	H,F	H,F	H,F		H,F,E
1,4-Dichlorobenzene	17		X	H,F	H,F	H,F		H,F,E
1,2-Dichloroethane	17		X	H,F	H,F	H,F		H,F,E
1,1-Dichloroethylene	17		X	H,F	H,F	H,F		H,F,E
2,4-Dinitrotoluene	27		X					
Hexachloroethane	none found							
Methyl Ethyl ketone	19		X					
Nitrobenzene	27		X					

Note: Refer to Figure 6 for the definitions of the Reaction Code entered on the squares of this worksheet.

Figure 2. Worksheet for determining hazardous waste compatibility.

X - Extremely reactive
E - Explosion
H - Heat
F - Fire

Waste A _____ Source _____

Waste B _____ Source _____

Name of Waste Evaluation _____ Date _____

WASTE A / WASTE B		Name	Arsenic	Barium	Cadmium	Chromium	Lead	Mercury	Selenium
		Reactivity Group No.	24	21,24,107	23,24	23,24	23,24	24	22,23,24
Name	Reactivity Group No.								
Pyridine	7		S	X	S	S	S	S	S
Vinyl chloride	17,103		P,H	X	P,H	P,H	P,H	P,H	P,H,E

Note: Refer to Figure 6 for the definitions of the Reaction Code entered on the squares of this worksheet.

Figure 2. Worksheet for determining hazardous waste compatibility.

- solubilization of toxic substances
P-- violent polymerization
X - extremely reactive
H - Heat

Waste A _____ Source _____

Waste B _____ Source _____

Name of Waste Evaluation _____ Date _____

WASTE A / WASTE B	Reactivity Group No.	Name	1,1,1-Trichloroethane 17	Trichloroethylene 17	Perchloroethylene 17	Methylene chloride 17	Trichlorotrifluoroethane (Freon) 17			
1,1,1-Trichloroethane	17									
Trichloroethylene	17									
Perchloroethylene	17			No	reactions					
Methylene chloride	17									
Trichlorotrifluroethane (Freon)	17									

Note: Refer to Figure 6 for the definitions of the Reaction Code entered on the squares of this worksheet.

Figure 2. Worksheet for determining hazardous waste compatibility.

Waste A _____ Source _____

Waste B _____ Source _____

Name of Waste Evaluation _____ Date _____

WASTE A / WASTE B	Reactivity Group No.	Name	Benzene 16	2,4-Dinitrotoluene 27	Methyl Ethyl Kentone 19	Nitrobenzene 27	Carbon Tetrachloride 17			
Benzene	16									
2,4-Dinitrotoluene	27									
Methyl Ethyl Ketone	19		No reactions							
Nitrobenzene	27									
Carbon Tetrachloride	17									
(representative of all 17)										

Note: Refer to Figure 6 for the definitions of the Reaction Code entered on the squares of this worksheet.

Figure 2. Worksheet for determining hazardous waste compatibility.

Waste A _____ Source _____

Waste B _____ Source _____

Name of Waste Evaluation _____ Date _____

WASTE A / WASTE B		Name	2,4-Dinitrotoluene	Methyl Ethyl Ketone	Nitrobenzene	Pyridine	Vinyl chloride	Chloroform		
Name	Reactivity Group No.	Reactivity Group No.	27	19	27	7	17	17		
2,4-Dinitrotoluene	27									
Methyl Ethyl Ketone	19									
Nitrobnzene	27									
Pyridine	17					-	H,GT	H,GT		
Vinyl chloride	17, 103					H,GT				
Chloroform	17					H,GT				

Note: Refer to Figure 6 for the definitions of the Reaction Code entered on the squares of this worksheet.

Figure 2. Worksheet for determining hazardous waste compatibility.

GT - Toxic gas generation
H --Heat

Waste A _____ Source _____

Waste B _____ Source _____

Name of Waste Evaluation _____ Date _____

WASTE A / WASTE B		Reactivity Group No. / Name	All 17 No's	(halogenated organics)						
Name	Reactivity Group No.									
Pyridine	7		H,GT							

Note: Refer to Figure 6 for the definitions of the Reaction Code entered on the squares of this worksheet.

Figure 2. Worksheet for determining hazardous waste compatibility.

H - Heat
GT - Toxic gas generation,

ATTACHMENT C-5

QUALITY ASSURANCE PROJECT PLAN

Attachment C-5

LABORATORY QUALITY ASSURANCE PROJECT PLAN

Solvent Reclamation Program
Detrex Corporation Solvents and Environmental Services Division

RECEIVED
JUL 9 1991
Waste Management Division

JULY 1991
REF. NO. 3467 (2)

CONESTOGA-ROVERS & ASSOCIATES

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APPENDIX A STANDARD OPERATING PROCEDURES (SOPs)

LIST OF ACRONYMS AND SHORT FORMS

Detrex	-	Detrex Corporation Solvents and Environmental ServicesDivision
FID	-	Flame Ionization Detector
GC	-	Gas Chromatograph
MDL	-	Method Detection Limit
NVR	-	Non-Volatile Residue
QA	-	Quality Assurance
QAO	-	Quality Assurance Officer
QAPP	-	Quality Assurance Project Plan
QC	-	Quality Control
USEPA	-	United States Environmental Protection Agency

1.0 INTRODUCTION

This Laboratory Quality Assurance Project Plan (QAPP) presents the policies, organization, objectives and specific quality assurance (QA) and quality control (QC) objectives designed to achieve the specific data quality goals associated with the Solvent Reclamation Program of Detrex. The purpose of the QAPP is to ensure that the sample results are accurate, precise and representative of the spent solvents analyzed. The analytical results are necessary for approving waste material acceptability for storage and subsequent reclamation at Detrex permitted TSD facilities.

2.0 PROJECT DESCRIPTION AND OVERVIEW

Detrex Corporation Solvents and Environmental Services Division operates numerous Treatment, Storage or Disposal (TSD) facilities which specialize in the sale of halogenated solvents and cleaning equipment and the reclamation (recycling) of solvents from spent solvents generated from various industries. Incoming samples of spent solvents from the TSD facilities are analyzed in order to determine the recoverable halogenated solvent content solely for processing purposes and to ensure that the waste materials are properly labeled and manifested for shipping purposes.

Before any hazardous waste material is accepted from a new customer, the waste is sampled by the facility and analyzed to ensure its characterization is acceptable based on the operating permit of the facility and to the ultimate recovery (recycling) operation. In addition to sampling, an effort is made to document the generation process of the waste to further characterize its composition.

Regardless of whether or not the facility accepts the waste for processing, all of the foregoing knowledge, including laboratory results, is kept in the customer's file for future reference and comparison with future shipments, if they become a regular customer.

2.1 TARGET COMPOUNDS

Target compounds for analysis will vary with the process that generated the spent solvents. Generally, a specific generation process will employ a specific solvent(s) in the operation. Consequently, the major constituents of the spent solvent samples are generally known prior to analysis and the analyses are performed for confirmatory and processing purposes. The target compounds for the spent solvents analyses are:

Compound	*Required Detection Limits*
1,1,1-Trichloroethane	0.5 %
Perchloroethylene	0.5 %
Trichloroethylene	0.5 %
Methylene Chloride	0.5 %
Trichlorotrifluoroethane (freon)	0.5 %

Other analyses that are performed are, percent solvent content, ignitability, other compounds, and percent non-volatile residue (NVR).

2.2 DATA USAGE

The principle usage of data is to confirm the permitted acceptability of the waste material for acceptance at the TSD facility and also to confirm the identification of the major constituents of the spent solvents and

determine the percentage of recoverable solvent. This information is used for processing and transportation purposes.

3.0 PROJECT DESCRIPTION AND RESPONSIBILITY

Figure 3.1 presents the key staff organization for the QAPP. A summary of each of the key responsibilities is presented below:

Project Manager,
Detrex Corporation Solvents and Environmental Services Division

- general overview of the project to ensure that the objectives are met
- overall responsibility for the solvent reclamation project

Operations Manger,
Detrex Corporation Solvents and Environmental Services Division

- general overview of the project operations
- responsible for the branch personnel sampling operations

Laboratory Manager,
Detrex Corporation Solvents and Environmental Services Division

- routine assessment of measurement systems for precision and accuracy
- data reduction, validation and reporting
- development of QA/QC reports to management
- coordinate and schedule laboratory analyses
- supervise in-house chain-of-custody
- review calculations
- prepare and approve analytical reports
- also serves as Quality Assurance Officer with the following responsibilities:
    - overview laboratory quality assurance

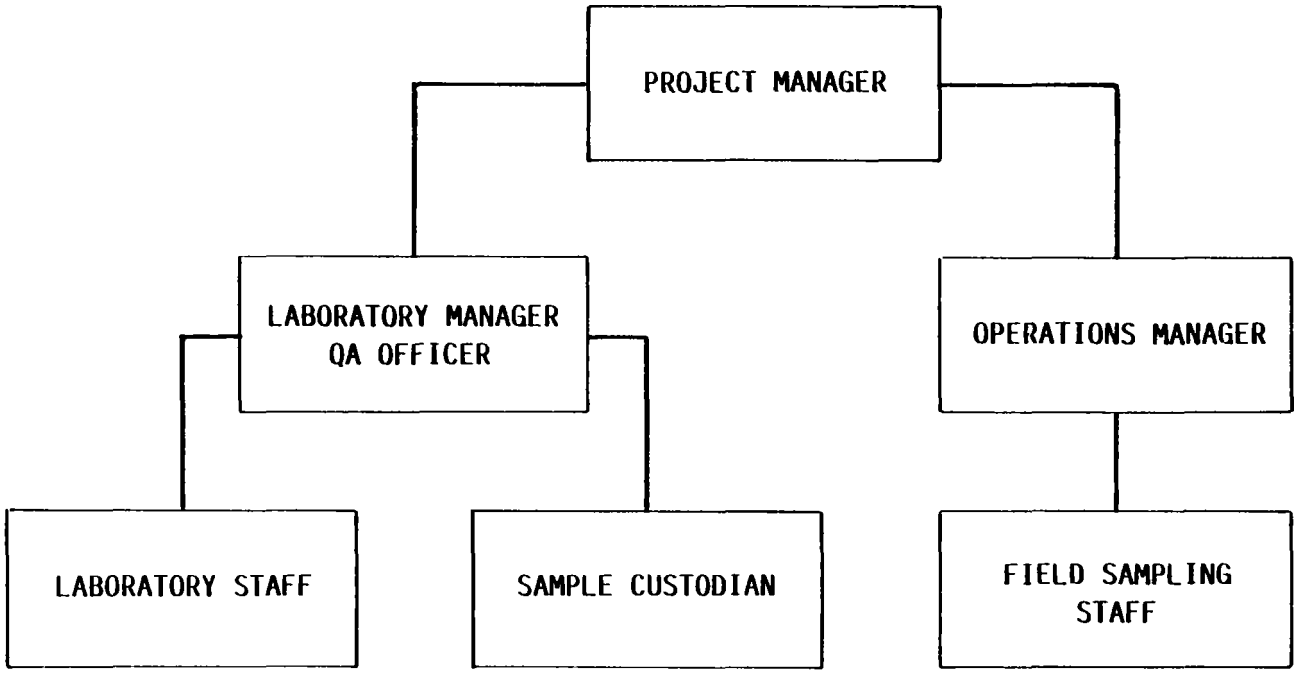

figure 3.1

STAFF ORGANIZATION
DETREX CORPORATION

CRA

3467-5/17/90-1-(C)-0

- also serves as Quality Assurance Officer with the following responsibilities:
    - overview laboratory quality assurance
    - overview QA/QC documentation
    - conduct detailed data review
    - implement laboratory corrective actions, as required
    - prepare laboratory Standard Operating Procedures (SOPs)
    - approve the QAPP

Laboratory Chemist

- sample extraction and instrumental analysis
- instrument maintenance and calibration
- standard preparation and documentation
- data reduction and calculations
- preparation of reagents
- wet chemical analytical determinations

Branch Personnel

- serve as field sampling personnel with the following responsibilities:
    - collection of representative samples
    - sample labeling and chain-of-custody implementation
    - performance of % solvent field determination
    - performance of ignitability test

4.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT OF DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS

The overall QA objective is to develop and implement procedures for sample handling, laboratory analyses and reporting that will provide accurate data. Specific procedures to be used for sample handling, calibration, laboratory analysis, reporting, quality control, audits, preventative maintenance and corrective actions are presented in subsequent sections of this QAPP. The purpose of this section, is to address the objectives of accuracy, precision, completeness, representativeness and comparability.

4.1 LEVEL OF QA EFFORT

Sample analysis are performed following the SOPs as provided in Appendix A. The levels of laboratory QA effort for the analyses are specified in the applicable SOPs provided in Appendix A.

4.2 ACCURACY, PRECISION AND SENSITIVITY OF ANALYSES

The QA objectives for precision, accuracy and sensitivity are to achieve the QC acceptance criteria for the analytical procedures. The precision and accuracy guideline requirements are specified in the SOPs as provided in Appendix A.

Precision and accuracy for the halogenated solvent and NVR determinations will be determined by using duplicate samples and spiked samples, respectively.

For duplicate sample analysis, at least one duplicate sample will be analyzed at a frequency of 1% of all samples, or one per day, whichever is more frequent. The relative percent difference (RPD) for each detected analyte will be calculated for use during data assessment and validation. An advisory limit of 30% for RPD will be used for detected analytes above the detection limit. RPD will not be calculated for non-detected analytes.

For reagent blank analyses, at least one reagent blank will be processed and analyzed for 1% of all samples analyzed, or one per day, whichever is more frequent. The concentration for each component will be calculated for later use during data assessment and validation.

The matrix spike sample analysis is used to provide information on the effect of the sample matrix on the distillation procedure. Matrix spike samples will be analyzed at a frequency of 1% of all samples, or one per week, whichever is more frequent. The individual component percent recoveries (%R) will be calculated and reported. The acceptance criteria for matrix spike samples are outlined in applicable SOPs presented in Appendix A.

Blank spike sample analysis is used to provide information on the accuracy of the method. Blank spike samples will be analyzed at a frequency of 1% of all samples, or one per week, whichever is more frequent. The individual component %R will be calculated and reported. The acceptance criteria for blank spike samples are outlined in applicable SOPs presented in Appendix A.

4.3 COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

It is expected that all analyses performed by the applicable SOPs will provide data meeting QC acceptance criteria for 80 percent of all samples analyzed. Any reason for variances will be documented. Corrective action procedures used to maximize the number of usable analyses are outlined in Section 14.0 of the QAPP.

The sampling procedure followed by facility personnel ensures that representative samples of the spent solvents will be obtained. Comparability of laboratory analyses will be ensured by the use of consistent units of concentration and standard operating procedures.

5.0 SAMPLE HANDLING, AND PRESERVATION TECHNIQUES

The following sections describe the sample handling techniques used in the project.

5.1 SAMPLE CONTAINERS, PRESERVATIVES AND HOLDING TIMES

Sample containers, preservatives and holding times are presented in Table 5.1.

5.2 SAMPLE LABELING AND DOCUMENTATION

The following protocols are employed by facility personnel during the sampling procedure:

1) Samples are labeled with the branch identification number, unique drum number(s), solvent type and sampling date.

2) A packing slip with branch identification, date shipped, drum numbers, solvent type, customer, analysis requested and percent solvent from field determination is completed for each shipment.

TABLE 5.1

SAMPLE CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR CONCENTRATED WASTE SAMPLES

Parameters	*Container*	*Preservative*	*Holding Time*
Volatile Organics	1 oz. glass with polyethylene-lined closure	none	14 days

3) The samples are packaged, sealed and delivered to the laboratory by common carrier at a minimum frequency of once per week.

4) Section 6.0 details the chain-of-custody procedure for the samples.

6.0 SAMPLE CUSTODY AND DOCUMENT CONTROL

This section details the procedures and protocols which are followed by facility personnel for the transport of samples to the appropriate laboratory. It is to be noted that for the Detroit, Michigan and Charlotte, North Carolina TSD facilities when analyses is to be conducted in the on-Site laboratory chain-of-custody procedures are not necessary.

6.1 CHAIN-OF-CUSTODY

A copy of the chain-of-custody form that accompanies any off-Site shipment of waste for analysis is provided as Figure 6.1.

6.2 SAMPLE DOCUMENTATION AND STORAGE IN THE LABORATORY

Sample receipt and log-in procedures are presented in the associated SOP in Appendix A.

FIGURE 6.1

DETREX CORPORATION

REQUEST FOR LABORATORY ANALYSIS

GENERATOR SUPPLIED INFORMATION (MUST BE COMPLETED)

Company Name _____ EPA ID No.: _____

_____ Salesman: _____

Address: _____ Comments: _____

_____ _____

Phone No.: (____) _____

_____ *RETURN* or *DISPOSE* of samples

Submitted by: Date

PROJECT NAME OR NUMBER _____ NUMBER OF SAMPLES _____

SAMPLE TYPE(S) _____

HAZARD CLASS _____

ANALYSIS(SES) REQUESTED _____

PLEASE LIST SAMPLE IDENTIFICATION NUMBERS ON REVERSE SIDE
(Column No. 2)

CHAIN OF CUSTODY

Relinquished By: Name	Date	Time	Received By: Name	Date	Time

FOR LABORATORY USE ONLY

Generator ID _____ Work Order No. _____

Sample results reported by: _____ Date _____ Time _____

Sample results reported to: _____

Sample results recorded: _____

Laboratory Book No. Page No.

Office Use LAB ID NO.	GENERATOR ID NO.	SAMPLE TYPE	ANALYSIS REQUESTED	COMMENTS

6.3 STORAGE OF SAMPLES

Upon completion of each job, all associated samples are packaged together and stored for a maximum period of 90 days prior to disposal.

6.4 SAMPLE DOCUMENTATION

Upon completion of analysis, a file for each job is assembled containing the following information:

- sample identification documents
- chain-of-custody forms, as appropriate
- raw data
- calculation
- originals of chromatograms
- originals of reports issued

7.0 CALIBRATION PROCEDURES AND FREQUENCY

The procedures indicated below are performed for all samples delivered to the laboratory for analysis. Specific procedures are presented in the applicable SOPs in Appendix A.

In general, the instruments are calibrated each day of sample analysis or the continuing calibration is verified using check standards. All calibration records are maintained by the laboratory for future reference.

7.1 GAS CHROMATOGRAPH CALIBRATION

Prior to analysis, the GC is calibrated as specified in the SOP in Appendix A.

7.2 PRECISION BALANCE CALIBRATION

The balance is initially calibrated at the factory and checked on a monthly basis. If the results of the periodic checks indicate a deviation from the expected value by more than 1%, recalibration by a certified technician must occur prior to use. The balance is cleaned and recalibrated by a certified technician at least yearly.

8.0 ANALYTICAL PROCEDURES

This section presents the analytical methods which are used by the laboratory to complete all required analyses.

8.1 HALOGENATED ORGANIC ANALYSIS

The method used for analysis of halogenated organics is a modification of Methods 8010 and 8015 for Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, EPA SW-846, 3rd edition, November 1986 (SW-846). The SOP is presented in Appendix A.

8.2 IGNITABILITY

The method used for ignitability testing is ASTM D 4982-89 "Standard Test Method for Flammability Potential Screening Analysis of Waste". The SOP is presented in Appendix A. The method used for analysis of flash point, which may be used on occasion to check ignitability results, is Method 1010 (Pensky-Marten Cup) for Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, EPA SW-846, 3rd edition, November 1986 (SW-846).

8.3 DETECTION LIMITS

Specific detections are highly matrix dependent. The detection limits for the analyses are presented in the SOPs in Appendix A.

9.0 DATA REDUCTION, VALIDATION AND REPORTING

Analytical data reduction and validation is performed under the direction of the laboratory QA officer (QAO). The QAO is responsible for assessing the data quality based on the QC results generated during the batch analyses. Figure 9.1 illustrates analytical data flow through the laboratory. Data reduction validation and reporting is conducted as detailed in the following steps:

- Raw data produced and checked by the responsible analyst is turned over for independent review by another analyst.

- The laboratory manager reviews the data for attainment of quality control criteria presented in the reference SOP.

- Upon completion of all reviews and acceptance of the raw data by the laboratory manager, a report is generated.

- The report and all supporting documentation is inspected by the QAO. Reanalysis of samples due to QC failure is determined and conducted.

- The final report is generated and signed by the laboratory manager.

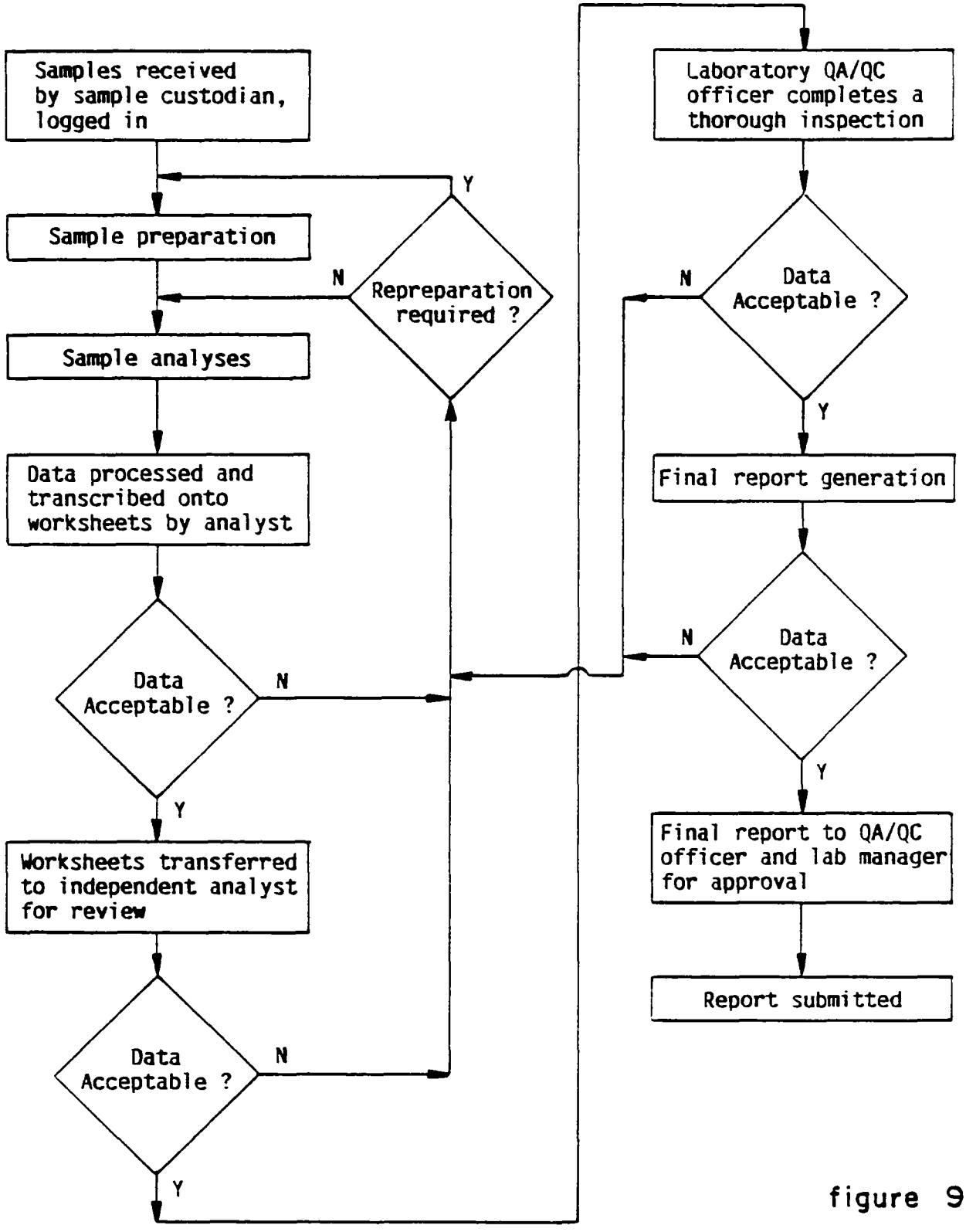

figure 9.1

ANALYTICAL DATA FLOW
DETREX CORPORATION

CRA

3467-5/17/90-1-(C)-0

10.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

The following sections present the internal quality control checks and frequency which are employed for laboratory measurements. Specific procedures related to internal laboratory QC samples (matrix spike, blank spikes, reagent blanks, duplicates) are detailed in the following subsections.

10.1 REAGENT BLANKS

A reagent blank will be analyzed at a frequency of 1%, or one per day, whichever is more frequent. The reagent blank, an aliquot of analyte-free solvent (chlorobenzene) will be carried through the entire analytical procedure.

10.2 MATRIX SPIKES

A matrix spike sample consisting of a sample fortified with a known concentration of analyte will be analyzed at a frequency of 1% or one per week, whichever is more frequent. Acceptance criteria and representative compounds are identified in the SOP presented in Appendix A. The results of the matrix spike samples are used to assess the accuracy of the method in a specific matrix.

10.3 BLANK SPIKE SAMPLES

A blank spike sample consisting on a synthetic matrix (oil/solvent mixture) fortified with all halogenated compounds of interest is analyzed at a frequency of 1% or one per week, whichever is more frequent. Acceptance criteria for these samples are presented in the applicable SOPs. The results of the blank spike are used to assess the accuracy of the method on a generic matrix typical of the spent solvent/oil mixture samples.

10.4 DUPLICATE SAMPLE ANALYSIS

Duplicate sample analysis are performed at a frequency of 1% or one per day, whichever is more frequent. This QC measure is performed to assess the analytical precision of the method and is applicable to the method as all samples will contain measurable concentrations of analyte(s).

10.5 SPLIT SAMPLES

Split samples are currently analyzed at a bi-yearly frequency. A typical sample is split and sent to an outside commercial laboratory with the results from both laboratories being compared and maintained on file.

10.6 BLIND PERFORMANCE EVALUATION SAMPLES

Blind performance evaluation samples are currently analyzed at a bi-yearly frequency. These consist of production samples split and analyzed by the QAO and the laboratory analysts. Results are compared and maintained on file.

11.0 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

For the purpose of internal evaluation, data from the project is evaluated on a continuing basis by the QAO over the duration of the project.

System audits are qualitative evaluations of all components of laboratory quality control measurement systems and are used to determine if the measurement systems are being used appropriately. The audits are conducted before the systems are operational and during the project. Such audits typically involve a comparison of the activities given the QA/QC plan described herein with activities actually performed.

The performance audit is a quantitative evaluation of the measurement systems used for a monitoring program. It requires testing the measurement systems with samples of known composition to evaluate precision and accuracy. The performance audit is accomplished by the review and statistical evaluation of QC sample results and is conducted and initiated by the QAO.

12.0 PREVENTIVE MAINTENANCE

Critical to the performance and minimization of downtime of all equipment, whether it be measurement or support, is proper maintenance. Preventive maintenance is performed as recommended by the manufacturer of the respective equipment. This maintenance will consist of, but is not limited to, the following minimum procedures at the specified time intervals.

12.1 PRECISION BALANCE

The balance will be cleaned and calibrated on a yearly basis by a certified technician, or more frequently should the need arise.

12.2 GAS CHROMATOGRAPH

Routine maintenance of the gas chromatograph includes daily checks of gas inventory, changes of septa as required by instrument performance, or at least weekly. Changes of injection port liners and clipping of the front end of the capillary column is performed as necessary. A maintenance contract with provisions for routine preventive maintenance visits is also maintained on the instrument.

13.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

13.1 QA MEASUREMENT QUALITY INDICATORS

13.1.1 Precision

Precision is assessed by comparing the analytical results between duplicate samples.

13.1.2 Accuracy

Accuracy is assessed by comparing a set of analytical results to the accepted or "true" values that would be expected. In general, matrix spike samples, duplicate samples and calibration check sample recoveries are used to assess accuracy.

13.2 STATISTICAL EVALUATIONS

In examination of data and determination of its precision and accuracy, standard statistical formulae will be used. Among these are the following:

1) <u>Mean of Measurements Generated by Each Sample Parameter (X)</u>

$$\overline{X} = \frac{1}{n} \left[\sum_{i=1}^{n} Xi \right]$$

where N = number of measurements

Xi = value of the measurement

2) <u>Standard Deviation of a Series of Individual Determination(s)</u>

$$S2 = \frac{\sum_{i=1}^{n} (Xi - x)2}{N - 1}$$

where N = number of measurements

Xi = value of the measurement

S = standard deviation

3) <u>Percent Relative Standard Deviation (% RSD)</u>

$$\%RSD = \left(\frac{s}{\overline{x}} \right) \times 100$$

s = standard deviation for a series of individual measurements

$\overline{x}$ = mean of the measurements

4) Percent Recovery

Percent recovery of spikes will be used to establish analytical accuracy and will be evaluated as follows:

$$\text{Matrix Spike Recovery} = \left(\frac{A-B}{C}\right) \times 100$$

where:

A = the analyte concentration determined experimentally from the spiked sample;

B = the background level determined by a separate analysis of the unspiked sample; and

C = the amount of the spike added.

5) Relative Percent Difference (RPD)

$$RPD = \frac{(|X1 - X2|)}{\left(\frac{X1 + X2}{2}\right)} \times 100$$

where:

X1 = first value, (i.e., MS value)

X2 = second value, (i.e., MSD value)

14.0 CORRECTIVE ACTION

The need for corrective action may be identified by system or performance audits or by standard QC procedures. The essential steps in the corrective action system will be:

- Checking the predetermined limits for data acceptability beyond which corrective action is required;

- Identifying and defining problems;

- Assigning responsibility for investigating the problem;

- Investigating and determining the cause of the problem;

- Determination of a corrective action to eliminate the problem (this may include reanalyses or resampling and analyses);

- Assigning and accepting responsibility for implementing the corrective action;

- Implementing the corrective action and evaluating the effectiveness;

- Verifying that the corrective action has eliminated the problem; and

- Documenting the corrective action taken.

For each measurement system, the QAO will be responsible for initiating the corrective action and the laboratory manager will be responsible for implementing the corrective action. The corrective action taken will depend upon the QA/QC criteria that did not meet the necessary criteria, and may range from qualifying the data to resampling. Corrective actions are further defined in the SOPs presented in Appendix A.

15.0 QUALITY ASSURANCE REPORT TO MANAGEMENT

Management will receive reports on the performance of the measurement system and the data quality at regular internals.

Minimally, these reports will include:

- Assessment of measurement quality indicators, i.e., data accuracy, precision and completeness;

- Results of system audits; and

- QA problems and recommended solutions.

The project QAO will be responsible within the organizational structure for preparing these periodic reports.

APPENDIX A

APPENDIX A
STANDARD OPERATING PROCEDURES

CONTENTS

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENT CONTENT IN SPENT SOLVENTS USING SPECIFIC GRAVITY METHOD

STANDARD OPERATING PROCEDURE FOR SAMPLE RECEIPT AND LOG IN

STANDARD OPERATING PROCEDURE FOR GLASSWARE CLEANING

STANDARD OPERATING PROCEDURES FOR THE EXTRACTION OF SPENT SOLVENTS

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENTS IN SPENT SOLVENTS BY GAS CHROMATOGRAPHY

STANDARD TEST METHOD FOR FLAMMABILITY POTENTIAL SCREENING ANALYSIS OF WASTE (ASTM D 4982-89)

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENT CONTENT IN SPENT SOLVENTS FROM DEGREASING OPERATIONS USING SPECIFIC GRAVITY METHOD

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENT CONTENT IN SPENT SOLVENTS FROM DEGREASING OPERATIONS USING SPECIFIC GRAVITY METHOD

1.0 SCOPE AND APPLICATION

The specific gravity method for correlating solvent percent and specific gravity was devised by Mr. Jim Goodrich, formerly laboratory supervisor of the Detrex Solvent Laboratory and currently a chemist in Detrex's Research Technologies International (RTI) division. The graphs relating specific gravity and solvent percent (volume) are non-empirical and are based on the following assumptions:

i) the non-volatile fraction of the waste oil can be approximated by an oil of specific gravity 0.9.

ii) the relationship between specific gravity and volume percent is linear.

iii) a sample of 0% solvent would have a specific gravity of 0.9, and a sample of 100% solvent would have a specific gravity of the solvent in question..

The specific gravity method is designed to be used a screening procedure only; quantitation is performed by a gas chromatograph analysis.

2.0 SUMMARY OF METHOD

A representative sample of the spent solvent/oil mixture is obtained and the specific gravity is determined. The percent solvent is determined by comparing the specific gravity of the mixture to that of the pure solvent.

3.0 INTERFERENCES

The determination is for screening purposes only and any materials that will interfere with the measurement of the specific gravity will interfere with the percent solvent determination.

4.0 APPARATUS AND MATERIAL

4.1 Glass Thief for Collection of Sample

4.2 Thermometer

4.3 Specific Gravity Hydrometer Set - 0.90 to 1.60 specific gravity range

4.4 Hydrometer Jar

5.0 REAGENTS

5.1 Rinse Solvent - Solvent of same type as being measured to rinse hydrometer jar

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in accordance to section 5.0 of the QAPP

7.0 PROCEDURE

7.1 Collect sample using the Glass Thief

7.2 Transfer sample to a container of sufficient size to allow for mixing.

7.3 Shake container for at least 30 seconds.

7.4 Place the thermometer and a hydrometer in the hydrometer jar.

7.5 Fill the hydrometer jar to within one inch of the top with the mixture to be tested.

7.6 If the hydrometer reads off-scale, replace it with a higher or lower range hydrometer as required.

7.7 Read the hydrometer to the nearest 0.01 SpG unit.

7.8 Pour sample back into original container.

7.9 Rinse hydrometer jar with solvent prior to collecting next sample.

7.10 Using the attached Solvent-Oil Mixture vs. Gravity Graph or Chart, for the appropriate solvent type, determine the weight per cent oil.

7.11 The solvent content is calculated as follows:

7.12 WEIGHT PER CENT SOLVENT = 100% - WEIGHT PER CENT OIL

8.0 QUALITY CONTROL

This procedure is for screening purposes only and no substantial quality control is required.

STANDARD OPERATING PROCEDURE
FOR SAMPLE RECEIPT AND LOG IN

STANDARD OPERATING PROCEDURE FOR SAMPLE RECEIPT AND LOG IN

1.0 SCOPE AND APPLICATION

The following describes the procedure for sample receipt and log in for the solvent reclamation program.

2.0 SUMMARY OF METHOD

Following collection, the samples are transported to the laboratory accompanied by chain-of-custody documentation. The samples are assigned a unique job number and unique sample numbers. All pertinent information is recorded in a bound, consecutively page-numbered logbook and scheduled for analysis.

3.0 PROCEDURE

3.1 Samples are delivered to the laboratory by means of common carrier and transferred to the sample custodian.

3.2 The sample custodian opens the package and removes the samples and associated chain-of-custody documentation. Any evidence of tampering with the sample package is recorded on the chain-of-custody document.

3.3 The contents of the package is compared against the chain-of-custody document and any discrepancies are noted.

3.4 The sample custodian assigns a unique job number and unique sample numbers to the samples.

3.5 The chain-of-custody document is completed and the job, sample numbers, and associated information is recorded in the sample log book.

3.6 An analytical request form is completed and the samples and copies of associated paperwork from each job is placed in a basket. The container is tagged with the job number and transferred to the appropriate analyst for analysis.

3.7 All original documentation is bound in a permanent book.

3.8 Upon completion of analysis, all samples from a job are stored together with the job tag for 90 days prior to disposal.

4.0 FORMS

Copies of forms and log books used during sample receipt and log in are attached for reference.

4-4-90
J-0111 7648-02-0585-American Precision-Freon-R&B

4-9-90
J-0112
7649-03-0234-Mag Products-111
7650-03-0235-Pan Rite-111
7651-03-0236-Electral-111
7652-03-0237-Turgeon & Sons-111
7653-03-0238-Caran Precision-111

4-9-90
J-0113
7654-04-3103-3105-TCE Great Neck Saw
7655-04-3106-3108-Perk-Gibson Tube Inc
7656-04-3109-DG-Union Broach
7657-04-3110-3114-TCE-Instrument Spec
7658-04-3115-3120-Manley Div KSG
7659-04-3117-DG Manley Div KSG
7660-04-3121-3126-TCE Textron Lycoming

4-9-90
J-0113
7661-04-3127-3130-TCE Textron Lycoming
7662-04-3131-TCE-Textron Lycoming
7663-04-3132-TCE-Textron Lycoming

J-0114
7664-13-Alumax-111-
7665-13-0217-TF-Southeastern Spec

4-10-90
J-0115
7666-11-4652-TRI-Liquid Waste Removal
7667-11-4653-TRI-Liquid Waste Removal
7668-11-4559-4560-TRI-US Engineering
7669-11-4556-4558-TRI-Brookside Corp
7670-11-4564-4569-Freon-Kingston Products
7671-11-4550-4555-Perk-Hoskins MFG
7672-11-4570-4571-Perk-Hoskins MFG
7673-11-4555-4557-TRI-IMC Corp.
4-10-90

PACKING LIST

5-16-90

RANCH 08-NC SALESMAN WATSON DATE SHIPPED 5/15 DATE RECVD ↑

J0210

~~AB ID~~	~~Drum~~ Nos.	Type Solv.	Drums Sampled No.	Total Wt.	Customer	% Solvent Est.	Lab
8210	2371-2380	1-1-1	10	4508	CONBRACO-P	45	
8211	2381-2385	1-1-1	5	2189	"	66	
8212	2432-2439	TRI	8	3891	R. BOSCH	52	
8213	2440-2447	TRI	8	3176	"	48	
8214	2448-2457	FREON	10	6166	"	93	
8215	2458-2467	FREON	10	4945	"	90	
8216	2468-2477	FREON	10	6098	"	96	
8217	2478	FREON	1	394	"	94	

Richard,

Results will be

Faxed to you by

1700 hrs. on 5-18-90

Phyllis

DETREX CORPORATION
SOLVENTS DIVISION

REQUEST FOR LABORATORY ANALYSIS

GENERATOR SUPPLIED INFORMATION (MUST BE COMPLETED)

Company Name _____ EPA ID No: _____

_____ Salesman: _____

Address: _____ Comments: _____

_____ _____

Phone No.: ( ) _____

_____ *RETURN* or *DISPOSE* of samples

Submitted by: Date

PROJECT NAME OR NUMBER _____ NUMBER OF SAMPLES _____

SAMPLE TYPE(S) _____

HAZARD CLASS _____

ANALYSIS(SES) REQUESTED _____

PLEASE LIST SAMPLE IDENTIFICATION NUMBERS ON REVERSE SIDE
(Column No. 2)

CHAIN OF CUSTODY

Relinquished By: Name	Date	Time	Received By: Name	Date	Time

==========

FOR LABORATORY USE ONLY

Generator ID _____ Work Order No. _____

Sample results reported by: _____ Date _____ Time _____

Sample results reported to: _____

Sample results recorded: _____

Laboratory Book No. Page No.

Office Use LAB ID NO.	GENERATOR ID NO.	SAMPLE TYPE	ANALYSIS REQUESTED	COMMENTS

STANDARD OPERATING PROCEDURE
FOR GLASSWARE CLEANING

STANDARD OPERATING PROCEDURE FOR GLASSWARE CLEANING

1.0 SCOPE AND APPLICATION

The following describes the procedure for cleaning glassware. The procedure is applicable to glassware used in processing spent solvent samples.

2.0 SUMMARY OF METHOD

Glassware is subjected to a solvent rinse, detergent wash, tap water rinse, deionized water rinse and allowed to air dry.

3.0 APPARATUS AND REAGENTS

3.1 Deionized Water - ASTM Type II

3.2 Detergent - Baxter Micro - All purpose laboratory cleaner

3.3 Mixed Solvent - Halogenated mixed reclaimed solvent

3.4 Brushes - Assorted flask and tube brushes

4.0 PROCEDURE

4.1 Remove oily residue by rinsing glassware with mixed solvent. Collect waste and allow to drain.

Note: This step is unnecessary for glassware not used for spent solvent mixtures.

4.2 Add detergent/water mixture and scrub vigorously to remove any remaining debris.

4.3 Drain and rinse thoroughly with tap water. Drain to remove excess tap water.

4.4 Rinse at least three times with deionized water. Water should sheet off glassware with a minimum of water droplets remaining on the inside surfaces. If not, repeat procedure.

4.5 Drain and allow to air dry prior to reuse.

STANDARD OPERATING PROCEDURES FOR THE EXTRACTION OF SPENT SOLVENTS

STANDARD OPERATING PROCEDURES FOR THE EXTRACTION OF SPENT SOLVENTS

1.0 SCOPE

This method describes a sampling preparation procedure for the determination of halogenated solvents in a spent halogenated solvent oil mixture.

2.0 SUMMARY OF METHOD

A sample of spent halogenated solvent waste is weighed in a tared, flat bottom flask. The flask is connected to a rotary vacuum evaporator and the solvent is vacuum distilled from the mixture, the weight of residue is determined, and the total non-volatile percentage calculated.

3.0 APPARATUS AND MATERIALS

3.1 Rotary vacuum evaporator with heating bath.

3.2 250 m*l* flat bottom flasks:

3.3 Precision Balance: capable of measuring to .005 $\pm$ 001 g.

3.4 Vacuum Apparatus: water aspirator.

4.0 PROCEDURE

Preheat water bath to 90°C. Pour approximately 30 m*l* of sample into a tared flat bottom flask, weigh and record initial weight. Attach flask to rotary vacuum evaporator, apply vacuum, lower flask into water bath and roto-evaporate solvent from solvent-oil sample until condensate ceases flowing from the condensor. Remove flask from evaporator, dry, reweigh and record. Remove (pour) distillate from receiving flask into clean sample container and store for analysis.

5.0 DETERMINATION OF % NON-VOLATILE RESIDUE

The calculation of % non-volatile residue is as follows:

$$\frac{R}{S} \times 100 = \% \text{ NV}$$

R=Mass of residue after roto-evaporation

S=Mass of waste solvent before roto-evaporation

6.0 The distillate is subsequently analyzed for individual halogenated solvents by gas chromatography.

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENTS IN SPENT SOLVENTS BY GAS CHROMATOGRAPHY

STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF SOLVENTS IN SPENT SOLVENTS BY GAS CHROMATOGRAPHY

1.0 SCOPE

1.1 This method is used for the determination of recoverable halogenated and non-halogenated solvents in spent solvent oil mixtures. Applicable analytes and their respective retention times are presented in Table 1.

1.2 The range of concentration determined by this method is 0.5 to 100% and is applicable only to concentrated organic matrices.

1.3 This method is based upon a gas chromatographic/flame ionization detection (GC/FID) procedure.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the samples are prepared for gas chromatography using the extraction procedure for waste solvent.

2.2 An aliquot of the extract is introduced into the gas chromatograph by direct injection. The solvents are separated by a capillary column and detected by a flame ionization detector.

TABLE 1
RETENTION TIMES FOR ORGANIC SOLVENTS

Compound	*Retention Time (minutes)*[1]
Methanol	2.83
Ethanol	3.16
Acetone	3.46
2-Propanol	3.51
t-Butyl alcohol	3.77
Freon	3.91
Methylene chloride	3.99
Nitromethane	4.45
Butylene oxide	5.19
Methylethylketone	5.22
sec-Butyl alcohol	5.32
Dioxolane	5.63
1,1,1-Trichloroethane	6.76
n-Butyl alcohol	7.69
Trichloroethylene	9.01
1,4-Dioxane	9.43
Methylisobutyl ketone	11.0
Toluene	12.4
Perchloroethylene	14.8
Cyclohexene oxide	17.8
o-Xylene	17.6
m-Xylene	18.2
p-Xylene	19.2

[1]Retention times are approximate and for illustration purposes only. Daily operating conditions will affect actual retention times.

3.0 INTERFERENCES

3.1 Potential interferences from the waste mixture are effectively reduced or removed by the vacuum distillation extraction procedure.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - Hewlett-Packard HP 5890 with Model 7673A autosampler and Model 3396 integrating recorder.

4.2 Column - Hewlett-Packard Ultra 2 (cross linked 5% phenyl methyl silicone gum) 50 meter x 0.32 mm ID with 0.52 µm film thickness

4.3 Detector - Flame Ionization Detector

4.4 Volumetric flasks - 10 ml Class A

4.5 Precision Balance - Capable of weighing 0.05 $\pm$ 0.01g

4.6 Pastuer Pipettes - 5 3/4" glass disposable

5.0 REAGENTS

5.1 Solvents - Chlorobenzene (ACS grade minimum with no interfering impurities)

5.2 Calibration Standards

Due to the high concentration range of this method, calibration standards may be prepared directly from the neat solvents.

5.2.1 Halogenated Solvents

ACS grade (99%+) solvents with no interfering peaks:

1,1,1-Trichloroethane
Trichloroethylene (trichloroethene)
Perchloroethylene (tetrachloroethene)
1,1,2-Trichloro-1,2,2-trifluoroethane (freon 113)
Methylene chloride

5.2.2 Non-Halogenated Solvents

ACS grade (99%+) solvents with no interfering peaks:

Methanol	n-Butyl alcohol
Ethanol	1,4-Dioxane
Acetone	Methylisobutyl ketone
2-Propanol	Toluene
t-Butyl alcohol	Methyl ethyl ketone

Nitromethane	Cyclohexene oxide
Butylene oxide	o-Xylene
sec-Butyl alcohol	m-Xylene
dioxolane	p-Xylene

5.2.3 Preparation of Standards

Calibration standards of the solvents are prepared at three concentration levels by weighing the appropriate mass of solvent in a tared 10 m*l* volumetric flask and diluting to the mark with chlorobenzene. Mixed standards may be prepared for the dilute standards. The procedure is:

- a clean, dry 10 m*l* volumetric flask is filled approximately half full with chlorobenzene. Allow the flask to stand approximately 10 minutes or until the wetted surfaces have dryed. Weigh the flask to the nearest 0.001 g.

- Using a Pastuer pipette, transfer the appropriate mass of solvent to the flask ensuring that the drops fall directly into the chlorobenzene without contacting the neck of the flask.

- Reweigh, dilute to the mark, stopper and mix by inverting the flask at least six times.

- Calculate the concentration in weight/volume percent from the net gain in weight. Correct for compound purity if purity is less than 98%.

- Repeat procedures for remainder of standards. For less concentrated standards, multiple compounds may be added.

- Transfer standards to the appropriate vials and cap with polyethylene lined closures.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sampling is completed by personnel at each Solvents Division facility according to protocols described in Section C of each facilities Part B Permit Application. Sample handling and preservation are outlined in Section 5.0 of the QAPP.

7.0 PROCEDURE

7.1 Extraction

The procedure for sample extraction presented in the Standard Operating Procedures.

7.2 Gas Chromatography Conditions

Initial oven temperature and holding time	35° - 6 minutes
Oven temperature program 1	4°C/minute
Second oven temperature and holding time	90°C - 0 minutes
Oven temperature program 2	10°C/minutes
Final oven temperature and holding time	180°C - 1 minute

Injector temperature	200°C
Detector temperature	250°C
Injector: Split-split ratio	1:100
Sample volume	1.0 μl
Carrier gas	Helium at 180 psi
Detector gas	Hydrogen at 18 psi
	Air at 45 psi

7.3 Calibration

7.3.1 External Standard Calibration Procedure

7.3.1.1 For each analyte of interest, prepare calibration standards at a minimum of three concentration levels by the procedure described in section 5.2.3.

7.3.1.2 Inject each calibration standard using the autosampler. Tabulate peak area response against the amount injected for

each compound of the standard. This ratio of area to amount injected is termed the calibration factor and is defined as:

$$\text{Calibration factor} = \frac{\text{Total Area of Peak}}{\%\ \text{component}}$$

If the percent relative standard deviation (%RSD) of the calibration factors is less than 20% over the working range, linearity throughout the origin is assumed and the average calibration factor may be used. If the %RSD is greater than 20%, then a calibration curve is calculated using linear regression analysis. The minimum acceptable correlation coefficient is 0.975.

7.3.1.3 The working calibration curve or calibration factor must be verified each working day by the inspection of one or more calibration standards. If the response for any analyte varies from the predicted response by more than ± 15%, a new calibration curve must be established for that analyte.

$$\%\ \text{difference} = \left(\frac{C_1 - C_2}{C_1}\right) \times 100$$

Where:

C_1 = Expected concentration

C_2 = Determined concentration

7.4 Gas Chromatographic Analysis

7.4.1 Inject 1.0 μl of the sample extract using the autosampler. If the response exceeds the linear range of the system, dilute the extract and reanalyze.

7.4.2 Calibrate the system (or confirm calibration) prior to conducting any analyses. A midlevel standard must also be injected at specific frequencies and at the end of the analytical batch. The determined concentration must be within $\pm$ 15% difference as compared to the expected concentration. If this criteria is exceeded, inspect the GC system to determine the cause and perform the necessary maintenance to correct the problem before recalibrating the system. All samples that were analyzed after the standard that exceeded the criteria are out of control and must be reanalyzed after the corrective action is taken.

7.4.3 Establish daily retention times for each analyte. Use absolute retention times for each analyte from the daily check standard as the midpoint for that day.

7.4.3.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time. No confirmation is necessary for the major degreasing solvents that are routinely determined. Compounds that are less frequently encountered will require an additional analytical technique for identification, if required. Tentative

identification is done more positively by spiking the sample with the suspected constituent and noting an increase in peak height. Sample would be sent to Detrex's RTI Lab in Detroit for positive ID, if necessary.

7.4.3.2 Validation of GC System Quantitative Performance

The midpoint calibration standard is used to evaluate this criterion. If any of the standards are outside of the daily retention time window, the system is out of control and corrective action will be necessary.

7.4.4 Sequence of Analysis

After the system has been calibrated and daily retention time calculated, the following sequence of analysis takes place:

1) Blank
2) 30 samples
3) Matrix Spike
4) Blank Spike
5) Duplicate
6) Calibration Check Standard(s)
7) Blank
8) 30 Samples

7.5 System Maintenance

These corrective actions may be required to remedy problems associated with poor response, shifting retention times, ghost peaks and unstable baselines.

7.5.1 System Bake-Out - To remedy problems associated with rising baselines and ghosts peaks, the chromatography system is baked out. This entails raising the oven temperature to the column conditioning temperature for two hours.

7.5.2 Septum Change - Unstable retention times and poor response may be attributed to a leaking injection port septum. Change septum per the maintenance manual guidelines.

7.5.3 Capillary Column maintenance - Ghost peaks and reduced response may be caused by dirty injection port inserts and front-end column contamination. Replace column insert with a clean-deactivated insert. Also, clipping the first few inches to a foot of the column may be necessary to remedy the problem.

7.5.4 Detector Maintenance-Reduced response may be caused by a dirty detector. Cool detector and clean as recommended in the maintenance manual. Recalibration of the system may be required after cleaning the detector.

7.6 Calculations

The concentration of each analyte in the sample is determined by calculating the peak response using the calibration factor or calibration curve determined in Step 7.3.1. The concentration of a specific analyte is calculated as follows:

$$\text{Concentration (\% wt/vol)} = \left(\frac{Ax}{cf} \right) \times D$$

where: Ax = Response for analyte in sample area units

cf = Calibration Factor - area units per unit concentrations

D = Dilution Factor (if any)

8.0 QUALITY CONTROL

8.1 Reagent Blanks - To demonstrate that the analytical system is not introducing analytes or interferences, reagent blanks consisting of chlorobenzene are processed with the samples. The blanks are carried completely through the extraction and measurement steps and are analyzed at a frequency of 1% of all samples or one per day; whichever is more frequent.

8.2 Accuracy Control

8.2.1 Matrix Spikes Samples - to demonstrate that the sample matrix is not affecting the accuracy of the measurement, matrix spike samples are analyzed. These samples consist of a sample aliquot that has been fortified with a known amount of analyte. The results are expressed as percent recovery (%R) of added analyte and are calculated by:

$$\%R = \left(\frac{SSR - SR}{SA} \right) \times 100$$

where SSR = Spike Sample result

SR = Sample result

SA = Spike added

Matrix spike samples are processed and analyzed at a frequency of one per week, whichever is more frequent. Due to the nature of the samples, only a specific sub set of analytes will be spiked into the sample.

8.2.2 Blank Spike Samples - To demonstrate the efficiency of the extraction and analytical procedures, blank spike samples are analyzed. These samples consist of an artificial matrix that is spiked with a known amount of all analytes of interest. The results are expressed as percent recovery (%R) of added analyte and are calculated by

$$\%R = \left(\frac{BSR - BR}{SA} \right) \times 100$$

where BSR = Blank Spike Result

BR = Blank result

SA = Spike added

Blank spike samples are processed and analyzed at a frequency of one per week.

8.3 Precision Control

To demonstrate the repeatability of the extraction and analytical procedures, duplicate sample analyses are performed. Due to the fact that there will be detectable analytes in every sample, this type of precision control is preferable to matrix spike, matrix spike duplicate analysis. Equal aliquots of a sample are processed and analyzed. The results are expressed as relative percent difference and are calculated by:

$$RPD = \frac{|SR_1 - SR_2|}{\left(\frac{SR_1 + SR_2}{2}\right)} \times 100$$

Duplicate samples are analyzed at a frequency of 1% of all samples or one per day, whichever is more frequent. RPD is calculated for %NVR and for detectable analytes.

where: SR_1 = sample result 1

SR_2 = sample result 2

8.4 Control Limits and Corrective Actions

8.4.1 Reagent Blanks

Reagent blanks should be free from interfering peaks and analytes of interest. Should there be evidence of contamination, the following should be investigated to locate and eliminate the source.

-Purity of solvent used for blank
-Cleanliness of glassware
-Use of lubricating grease on distillation apparatus
-Adequate rinsing of GC syringe
-Carryover from previous sample analysis
-Contaminated GC system

If the problem is determined to be isolated only to the reagent blank (in the case of carryover) no corrective action is required. Should the problem be determined to be systematic, re-extraction and analysis of the samples associated with the reagent blank must take place. Documentation of corrective actions taken is recorded in a bound laboratory notebook.

8.4.2 Matrix Spike Samples

Recovery limits for spike compounds are listed below:

1,1,1-Trichloroethane	75-125%
Trichloroethylene	75-125%
Perchloroethylene	75-125%
Freon	65-125%
Methylene chloride	75-125%

Should recovery limits be outside the acceptance criteria, the following should be investigated or conducted:

-Re-analysis of the matrix spike sample
-Evaluation of results of sample analysis. Concentrated samples may cause the spiked sample to be outside the linear range of the instrument. Dilute and re-analyze.
-Sample homogeneity
-Loss of instrument calibration - analyze a check standard for calibration verification

Should re-analysis of the matrix spike sample still indicate an out-of-control situation, a new sample will be chosen for matrix spike analysis. If analysis still yields out-of-control results, a thorough investigation must be conducted and the finding documentated. All samples processed from the last in-control point must be reprocessed and re-analyzed.

Should results of matrix spike sample analysis indicate an isolated problem associated with the blank spike sample, re-extraction and re-analysis is not required. In all other cases, re-extraction and re-analysis from the last in-control sample must occur.

8.4.3 Blank Spike Samples - Recovery limits for blank spike compounds are listed below:

1,1,1-Trichloroethane	75-125%
Trichloroethylene	75-125%
Perchloroethylene	75-125%
Freon	65-125%
Methylene chloride	75-125%

Should the above recovery limits be exceeded, the following should be investigated or conducted:

-Re-analysis of the blank spike sample
-Contamination of distillation due to loss of vacuum or inappropriate bath temperature
-Loss of calibration of GC. A check standard should be analyzed.

8.4.4 Replicate sample analysis - RPD limits for NVR and detected compounds are 30%. Should this criterion be violated, the following should be investigated or conducted.

-Re-analysis of samples
-Contamination of distillation and GC systems
-Homogeneity of sample matrix
-Inefficient distillations

Should re-analysis of samples not elicit in-control results, a new sample from the batch will be chosen for replicate analysis. If the results of the new replicates still indicate an out-of-control situation, a thorough investigation must be conducted and the findings documented. All samples processed from the last in-control point must be re-extracted and re-analyzed.

9.0 METHOD PERFORMANCE

At this time, the performance of the method is still being evaluated.

10.0 REFERENCES

1) Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, EPA SW-846 Methods 8000, 8010 and 8015.

2) 1989 Annual Book of ASTM Standards, ASTM Volume 15.05 Methods D 3742 and D-3447, Volume 14.01.

STANDARD TEST METHOD FOR FLAMMABILITY POTENTIAL SCREENING ANALYSIS OF WASTE (ASTM D 4982-89)

Designation: D 4982 – 89

Standard Test Methods for Flammability Potential Screening Analysis of Waste[1]

This standard is issued under the fixed designation D 4982; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods are used to indicate the fire-producing potential of wastes. The following test methods can be applied to waste liquids, sludges, semisolids, or solids:

	Sections
Test Method A—Test Sample Exposed to Heat and Flame	11 to 13
Test Method B—Test Sample Exposed to Spark Source	14 to 15

1.2 These test methods should be used to measure and describe the properties of materials, in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials under actual fire conditions. However, results of these tests may be used as elements of a fire risk assessment that takes into account all of the factors that are pertinent to an assessment of the fire hazard of a particular end use.

1.3 These test methods are designed and intended as preliminary tests to complement the more sophisticated quantitative analytical techniques that may be used to determine flammability. These test methods offer, to the user, the option and the ability to screen waste for potentially hazardous flammability potential when the more sophisticated techniques are not available and the total waste composition is unknown.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard information is given in Section 6 and 13.4.3.1.

2. Referenced Documents

2.1 *ASTM Standard:*

D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester[2]

2.2 *Other Standard:*

Prudent Practices for Handling Hazardous Chemicals in Laboratories[3]

3. Terminology

3.1 *Description of Term Specific to This Standard:*

3.1.1 *screening analysis*—a *preliminary* qualitative or semiquantitative test developed from classical qualitative and quantitative techniques that is designed to efficiently give the user specific information about a waste that will aid in determining waste identification, process compatibility, and safety in handling.

4. Summary of Test Methods

4.1 *Method A*—A test sample is exposed to heat and flame. The sample is reported as having a positive or negative flammability potential as described in the test procedure.

4.2 *Method B*—Sparks from a flint lighter are introduced to the vapor space immediately above a representative sample of a waste, and observation is made for a flash in the vapor space or ignition of the sample. A flash in the vapor space or ignition and burning of the waste indicates a positive flammability potential at ambient temperature.

5. Significance and Use

5.1 These test methods are intended for use by those in the waste management industries to aid in identifying the flammability potential or waste materials.

6. Hazards

6.1 Avoid inhalation and skin or eye contact, or both, of any hazardous materials.

6.2 Standard laboratory hygiene practices should be followed when conducting these tests.

6.3 All tests must be performed in a laboratory hood equipped with an exhaust intake along the front edge of the work area to collect any heavier-than-air vapors generated.

6.4 The analyst must wear flame resistant oven gloves.

6.5 Waste containing or suspected of containing highly volatile organics or peroxides should be tested using a much smaller sample than that used in 13.2.

7. Sampling

7.1 Sample containers must be kept tightly sealed until tested.

7.2 Samples should be analyzed as soon as possible after collection.

7.3 If necessary, allow the sample to come to room temperature in a tightly sealed container. For example, frozen material should be allowed to thaw completely.

8. Report

8.1 Report the following information:

8.1.1 Sample identification,

8.1.2 Date of test,

8.1.3 Sample classification: positive or negative, and

8.1.4 Reference to the procedure applied.

[1] These test methods are under the jurisdiction of ASTM Committee D-34 on Waste Disposal and is the direct responsibility of Subcommittee D34.02 on Physical and Chemical Characterization.

Current edition approved Nov. 10, 1989. Published December 1989.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] National Research Council, National Academy Press, Washington, DC, 1981.

9. Quality Control

9.1 Quality control check samples and duplications should be performed at an action level specified by the laboratory and at an appropriate frequency.

9.2 Flammability standards should be maintained for analyst training and as reference guides. Examples are given in the following table.

Flammability Potential Screening Analysis

Methyl alcohol (methanol)	Difficult-to-see flame; flammable at or below room temperature.
Glacial acetic acid	Flammable at temperatures above normal room temperature.
Hendecane (n-undecane)	Flammable at temperatures higher than glacial acetic acid.
Kerosene (Fuel Oil No. 1)	Flammable over a range of temperatures higher than normal room temperature.
p-Xylene	Flammable at or above room temperature; freezes at 13°C. A spiked soil sample frozen at 0°C demonstrates interference.
Chlorofluorocarbon 113 (1,1,2-trichloro-1,2,2-trifluoroethane)	Warning: phosgene formation likely; see 13.4.3.1.

10. Precision and Bias

10.1 No statement is made about either the precision or bias of these test methods since the result merely states whether there is conformance to the criteria for success specified in the procedure.

TEST METHOD A—EXPOSURE TO HEAT AND FLAME

11. Interferences

11.1 Drafts in the laboratory fume hood where the test is performed could cause excessive cooling and false negative results. A properly operating fume hood with a face velocity of 150 ft/min should provide consistent, usable results.

11.2 Ignition sources that provide excessive heating rates alone or combined with a very small test portion may obscure results. That is, the sample may be decomposed, sintered, fused, evaporated, or otherwise consumed before positive or negative evidence of flammability is observed. The use of sufficiently large test portions and of heating rates typical of a bunsen burner should resolve this problem.

11.3 An inadequate source of heating could limit the volatilization of flammable components and provide false negative results.

11.4 Difficult-to-observe flames resulting from the burning of certain compounds (for example, methanol) could provide false negative results. If the presence of these compounds is suspected, the presence of flammability might be confirmed by the insertion of a watchglass just above the test material. The watchglass is then examined for products of combustion (for example, moisture and soot).

NOTE 1—The watchglass method cannot distinguish between vaporized water or water produced by combustion.

11.5 An improperly adjusted gas burner (for example, with an insufficiently aerated flame) could introduce raw gas into or immediately above the sample. This raw gas could briefly support a flame after the source of ignition is removed, providing the appearance of a flammable sample and a false positive result.

12. Apparatus

12.1 *Gas Burner*, (for example, a bunsen burner) with an adjustable air shutter and an adjustable gas orifice is needed. The gas burner and fuel supply line must be appropriate to the gas supplied: natural gas, artificial gas (including propane and butane), or liquified petroleum gas (LP gas or LPG). Where a gas supply line cannot be provided, a propane torch may be substituted.

12.2 *Lighter*, (for example, piezo lighter) for burner is required.

12.3 *Aluminum Weighing Boats* or other non-flammable containers are needed.

12.3.1 **Caution**—Weighing boats of material other than aluminum should be used if the testing materials react with aluminum, for example, caustics.

12.4 *Watchglass*.

12.5 *Large Beaker, Tongs, Asbestos-Free High-Temperature Gloves or Mittens*, or other apparatus as needed to extinguish burning materials.

13. Procedure

13.1 Light a gas burner and adjust to a typically blue flame that is not readily blown out. A yellow flame easily affected by drafts indicates insufficient air (the air:fuel ratio is too low). A sharp, blue flame is good. (If the flame rises above the burner head, is very difficult to light, or tends to extinguish itself, indicates that too much air or too much air and fuel are being supplied to the burner.)

13.2 Place a sufficient amount (approximately 5 g) of a test sample in an aluminum weighing boat or other nonflammable container.

13.3 Using a gas burner, hold the flame immediately above the test sample for 2 to 3 s without touching the visible flame to the sample.

13.3.1 If ignition (a flash or burning) is observed before or after the source of ignition (the flame of the burner) is removed, the sample is said to have a positive flammability potential. A positive result may require further investigation (see 13.4.1).

13.3.2 The confirmation of flammability may require the use of a watchglass (see 11.4).

13.3.3 If there is no ignition, proceed to 13.4.

13.4 Using a gas burner, briefly (for at least 15 s) apply the flame to the sample in an attempt to ignite the sample.

13.4.1 If the sample ignites, the sample is said to have a positive flammability potential.

13.4.1.1 When more accurate waste characterization is necessary, liquid samples may be quantified using a closed-cup flash point tester.

13.4.1.2 Solids with a positive flammability potential should be further investigated.

13.4.2 If the sample decomposes, boils (if a liquid), or otherwise fails to ignite after at least 15 s of continuous sample heating by the burner flame, the flammability potential is reported as negative.

13.4.3 Halogenated solvents typically give off visible vapors that may result in a false positive flammability potential.

NOTE 2: Warning—Phosgene, an extremely toxic gas, is a combustion product of halogenated compounds burned in air.

13.5 Shut off the gas burner when not in use. Extinguish a

burning sample by setting an aluminum weighing boat or watchglass atop the one containing the burning sample, (or invert a spoutless beaker over the sample container and all). Use of tongs or high temperature gloves or mittens may be necessary to handle the equipment.

TEST METHOD B—EXPOSURE TO SPARK SOURCE

14. Apparatus

14.1 *Oven Gloves.*

14.2 *Flint Lighter*, the type typically used to light an air/acetylene torch is required.

14.3 *Disposable 250-mL Beaker*, of plastic is required.

14.4 *Watchglass*, 100 mm.

14.5 *Metal Vessel* (with lid), of adequate depth and diameter to contain beaker and watchglass is needed.

14.6 *Thermometer.*

15. Procedure

15.1 Place approximately 100 mL of the representative sample of the material to be tested into the plastic beaker (see 7.3).

15.2 Place the plastic beaker in the steel vessel, cover the beaker with the watchglass, and allow to stand at ambient conditions for 5 min.

15.3 Record the ambient temperature.

15.4 Remove the watchglass, place the igniter immediately above the waste and strike it several times to produce sparks.

15.5 If the material does catch fire and burn, extinguish the flames by immediately placing the lid on the steel vessel, thus smothering the fire and report as positive flammability potential.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

ATTACHMENT C-6

STANDARD OPERATING PROCEDURE FOR

WASTE SAMPLING

STANDARD OPERATING PROCEDURE FOR

WASTE SAMPLING

STANDARD OPERATING PROCEDURE
WASTE SAMPLING

This section describes in detail the procedures that is to be followed for sampling the containers of spent organic solvents.

1. SAFETY CONSIDERATIONS

- proper ventilation and dermal protection is provided for the sampling operation

- any built-up pressure within a container is slowly released by gently cracking the bung

2. SAMPLING EQUIPMENT

Materials and equipment required for sampling are as follows:

1) 1 oz. narrow-mouth glass bottle with polyethylene-lined closure.
2) sample label.
3) 4-foot by 3/4 inch ID sampling thief.
4) bung wrench (non-sparking).
5) packing slip and chain-of-custody form (for off-site analyses).

3. DRUM SAMPLING

3.1 Discrete Liquid Concentrated Waste Samples

The following procedure shall be adhered to during the sampling of individual drums.

1) Remove bung using non-sparking bung wrench.
2) Slowly insert thief to the bottom of the drum or until a solid layer is encountered. About one foot of glass thief should extend above the drum.
3) Allow the waste in the drum to reach its natural level in the thief.
4) Cap the top of the thief with gloved thumb.

5) Carefully remove the thief from the drum and insert the uncapped end in a graduated beaker.
6) Gently pour appropriate sample volume into sample jar
7) Label sample.

The following procedures shall be adhered to during the sampling of bulk waste shipments.

1) Open access chamber into tanker truck.
2) Slowly insert thief to the bottom of the waste.
3) Allow the waste in the tanker to reach its natural level in the thief.
4) Cap the top of the thief with gloved thumb.
5) Carefully remove the thief from the tanker and insert the uncapped end in a graduated beaker.
6) Gently pour appropriate sample volume into sample jar.
7) Label sample.

3..2 Composite Sampling

The following procedure shall be adhered to during composite sampling. A maximum of ten drums may be composited. Only drums of the same waste type from the same generator may be composited.

1) Collect a drum sample from each drum according to the procedure described above.
2) Place sample from each drum to be composited into the same graduated beaker.
3) Mix entire composite gently and pour appropriate sample volume into sample jars.
4) Label sample.

4. CHAIN-OF-CUSTODY

For all analysis conducted off-Site, a chain-of-custody form (sample attached) shall be completed.

DETREX CORPORATION

REQUEST FOR LABORATORY ANALYSIS

GENERATOR SUPPLIED INFORMATION (MUST BE COMPLETED)

Company Name: _____ EPA ID No.: _____

_____ Salesman: _____

Address: _____ Comments: _____

_____ _____

Phone No.: (____) _____

_____ *RETURN* or *DISPOSE* of samples

Submitted by: Date

PROJECT NAME OR NUMBER _____ NUMBER OF SAMPLES _____

SAMPLE TYPE(S) _____

HAZARD CLASS _____

ANALYSIS(SES) REQUESTED _____

PLEASE LIST SAMPLE IDENTIFICATION NUMBERS ON REVERSE SIDE
(Column No. 2)

CHAIN OF CUSTODY

Relinquished By: Name	Date	Time	Received By: Name	Date	Time

FOR LABORATORY USE ONLY

Generator ID _____ Work Order No. _____

Sample results reported by: _____ Date _____ Time _____

Sample results reported to: _____

Sample results recorded: _____

Laboratory Book No. Page No.

Office Use LAB ID NO.	GENERATOR ID NO.	SAMPLE TYPE	ANALYSIS REQUESTED	COMMENTS

SECTION D

PROCESS INFORMATION

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PROCESS INFORMATION

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Date: 09/03/91
Revision: 91-2

LIST OF PROCESS EQUIPMENT

I.D. No.	*Equipment Name*	*Description*
1.	Generator	Clayton Model E-100 Steam Generator. Unit burns natural gas to produce steam at the rate of 3450 lbs./hr. at 100 psig.
2.	Air Compressor	
3.	20,000 gal. Product Tank	20,000 gallon carbon steel storage tank for storage of 1,1,1 Trichloroethane.
4.	10,000 gal. Product Tank	10,000 gallon carbon steel storage tank for storage of Trichloroethylene
5.	4,500 gal. Product Tank	4,500 gallon carbon steel storage tank for storage of Perchloroethylene.
6.	4,500 gal. Product Tank	4,500 gallon carbon steel storage tank for storage of Trichloroethylene
10 A/B	2 - 600 gal. Receiver Tanks	Used for receiving product from Detrex stills. (Operated at atm. pressure).
12.	2,300 gal. Hazardous Waste tank storage tank	2,300 gallon carbon steel storage tank used for storage of F001 or F002 material prior to processing
13.	350 gal. Detrex Still	Detrex Model S-350. Used for recovering chlorinated solvents from spent solvents from degreasing operations (F001 material) via distillation. This unit can process approximately 2,000 gallons/day.
14.	350 gal. Detrex Still	Detrex Model S-600. Used for recovering chlorinated solvents from spent solvents from degreasing operations (F001 material) via distillation. This unit can process approximately 2,000 gallons/day.
15.	DCI Still	DCI Model Dyna-1-100 Solvent Recovery Still. Used to recover chlorinated solvents from still bottoms from recovery of same (F002 material) via live steam injection. This unit can process approximately 100 gallons per hour.
16 A/B	Drying Columns	Detrex Dual Column Drier. Used to remove water from recovered product (solvent) via adsorption.

Date: 09/03/91
Revision: 91-2

LIST OF PROCESS EQUIPMENT

I.D. No.	*Equipment Name*	*Description*
17.	5,000 gal. Still Bottom Tank	5,000 gallon carbon steel storage tank. Used for temporary accumulation of still bottoms from recovery of chlorinated solvents (F002 material).
20.	2,500 gal. Holding Tank	2,500 gallon 316 stainless steel storage tank used for storage of reclaimed solvent.
21.	4,500 gal. 1,1,1 Trichloroethane	4,500 gallon carbon steel storage tank for storage of 1,1,1 Trichloroethane.
22.	4,500 gal. Hazardous Waste Storage Tank	4,500 gallon carbon steel storage tank used for temporary storage of F001 or F002 material prior to being processed by Detrex stills.
24.	DCI Still	DCI Model Dyna-1-500 Solvent Recovery Still. Used to recover chlorinated solvents from still bottoms from recovery of same (F002 material) via live steam injection. This unit can process approximately 500 gallons per hour.
25.	3,000 gal. Holding Tank	3,000 gallon 316 stainless steel storage tank used for storage of reclaimed solvent.
26	SVRM - Carbon Absorption Unit	
27.	Drum Filling Station	Product Drumming Station. Used for filling 55-gallon drums with product. Unit can fill approximately 30 drums per hour and is operated as necessary.
28.	Product Blending Vessel	550 gallon carbon steel vessel utilized for product blending.

Date: 09/03/91
Revision: 91-2

LIST OF PROCESS EQUIPMENT

I.D. No.	*Equipment Name*	*Description*
1.	Generator	Clayton Model E-100 Steam Generator. Unit burns natural gas to produce steam at the rate of 3450 lbs./hr. at 100 psig.
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4.	10,000 gal. Product Tank	10,000 gallon carbon steel storage tank for storage of Trichloroethylene
5.	4,500 gal. Product Tank	4,500 gallon carbon steel storage tank for storage of Perchloroethylene.
6.	4,500 gal. Product Tank	4,500 gallon carbon steel storage tank for storage of Trichloroethylene
10 A/B	2 - 600 gal. Receiver Tanks	Used for receiving product from Detrex stills. (Operated at atm. pressure).
12.	2,300 gal. Hazardous Waste tank storage tank	2,300 gallon carbon steel storage tank used for storage of F001 or F002 material prior to processing
13.	350 gal. Detrex Still	Detrex Model S-350. Used for recovering chlorinated solvents from spent solvents from degreasing operations (F001 material) via distillation. This unit can process approximately 2,000 gallons/day.
14.	350 gal. Detrex Still	Detrex Model S-600. Used for recovering chlorinated solvents from spent solvents from degreasing operations (F001 material) via distillation. This unit can process approximately 2,000 gallons/day.
15.	DCI Still	DCI Model Dyna-1-100 Solvent Recovery Still. Used to recover chlorinated solvents from still bottoms from recovery of same (F002 material) via live steam injection. This unit can process approximately 100 gallons per hour.
16 A/B	Drying Columns	Detrex Dual Column Drier. Used to remove water from recovered product (solvent) via adsorption.

Date: 09/03/91
Revision: 91-2

LIST OF PROCESS EQUIPMENT

I.D. No.	*Equipment Name*	*Description*
17.	5,000 gal. Still Bottom Tank	5,000 gallon carbon steel storage tank. Used for temporary accumulation of still bottoms from recovery of chlorinated solvents (F002 material).
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22.	4,500 gal. Hazardous Waste Storage Tank	4,500 gallon carbon steel storage tank used for temporary storage of F001 or F002 material prior to being processed by Detrex stills.
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28.	Product Blending Vessel	550 gallon carbon steel vessel utilized for product blending.

SECTION D

PROCESS INFORMATION

This section provides specific process information for the hazardous waste container and tank storage areas at the Detrex Corporation facility in Detroit, Michigan. The details are provided pursuant to Michigan Act 64 Rule 299.9504(1)(g), (2), and (3) which incorporates by reference 40 CFR §270.15 and 16. The applicable section(s) of the federal regulations (40 CFR) is referenced where applicable.

The hazardous waste management operations include container storage and tank storage. Wastes are received in containers or bulk shipments. The wastes are processed through the recovery (recycling) system which primarily consists of distillation followed by drying. The recovered solvent product is drummed or stored in product tanks for re-sale and the still bottoms remaining after distillation are transferred to a 5,000 gallon generator accumulation tank. The still bottoms are accumulated for less than 90 days prior to shipment off site to a permitted facility.

A facility plan is presented in Attachment D-1. This drawing locates all process operational units within the building. A map key is provided which cross-references identification numbers on the plan to the appropriate equipment identification. Attachment D-2 provides copies of available engineering plans for the facility. The building was apparently constructed in 1946 and has been occupied by Detrex since 1966. Within Attachment D-2 are the following:

1) A Plan of Survey prepared by Warner, Cantrell and Padmos, Inc. This survey plan shows an accurate layout of the facility. The survey plan also provides several structural details in cross-section view to illustrate secondary containment features;

2) New Gold Shield Reclamation Addition, prepared by Detrex Corporation provides some engineering details from a 1966 renovation which provided new office space at the southwest corner of the building and a new product storage tank pad; and

3) An engineering certification of the above-referenced plans as well as the tank drawings provided in Attachment D-6.

D-1 CONTAINERS [40 CFR §270.15]

Hazardous wastes that are received at the Detrex solvent recovery facility in DOT-approved containers (typically 55-gallon drums) are placed in a hazardous waste container storage area prior to recycling. The location of the container storage area is shown in Attachment D-1. An additional container storage area is present in the facility, however, only virgin solvent products are stored in this area.

The hazardous waste container storage area is delineated by yellow lines painted on the concrete floor. In addition, each container is labelled according to its contents and manifest information. Only containers of the same type of waste (i.e. Perchloroethylene) are stored in the same row. Product labels are completely different to distinguish between product and hazardous waste containers.

D-1a(1) Description of Containers [40 CFR §264.171 and .172]

Virgin solvent is sold by Detrex Corporation to customers in new 16 or 18 gauge, DOT 17E, 55-gallon drums or other DOT-approved containers. When the solvent is returned to Detrex in waste form, it is usually returned in the original container in which the virgin material was shipped. If it is not returned in the original container, it must be returned in a container which meets the following:

1. Not have any leaks;

2. Not have excessive corrosion or rust build up which could, upon handling, become a leak;

3. Not be excessively dented such that the usable volume is less than the nominal capacity of the container (i.e. 55 gallons for 55-gallon drums);

4. Be properly sealed with suitable bung;

5. Be properly labelled and manifested; and

6. Meet other appropriate DOT requirements for shipment of hazardous material and/or hazardous wastes.

Since Detrex delivers virgin solvents to customers in its own trucks, it is common practice to pick up the waste material at the same

time. Detrex they are able to check each container to see that the criteria outlined above are met. Containers not meeting this criteria are not accepted.

If waste containers are received at the facility by other than Detrex's truck, the drums are inspected upon arrival, to ensure their conformance with the above listed requirements.

While stored at the facility, should a container incur a breach of integrity, they thereof will be immediately transferred to a container in good condition.

While stored at the Detrex facility, all hazardous waste containers are properly labelled.

D-1a(2) Container Management Practice [40 CFR §264.173]

To ensure that no spillage of hazardous waste material occurs in shipment from a customer facility to the Detrex facility or within the Detrex facility, all containers must meet the requirements specified previously. The containers remain tightly sealed during storage in the container storage area and are opened only when the waste material is to be tested or being transferred to the process equipment.

Drums are stored in the vertical position and are situated in rows with a minimum 2 1/2-foot aisle space to allow for inspection. If necessary, drums are stacked up two layers high. Each row of drums is placed on wooden pallets to elevate the drums off the floor. Attachment D-3 provides a facility plan illustrating typical container storage arrangement under maximum inventory. Actual storage varies depending on inventory size and waste type. Attachment D-3 also illustrates a cross-sectional view of a row showing typical stacking arrangement. Should other types of DOT-approved containers to accepted for storage, they will also be placed on wooden pallets and aisle space shall be maintained.

Drums are transferred within the building by a forklift with a drum handling attachment. Hand drum trucks may also be used to transport drums short distances. Other containers are typically transferred with the forklift.

There are no ignitable, reactive or incompatible hazardous wastes handled within the hazardous waste container storage area.

Hazardous waste containers are distinguished from virgin product containers by proper hazardous waste labelling and segregation within the facility. Hazardous wastes are labelled in order to identify the contents to ensure proper transfer to the recycling operational system as each waste type is processed separately. Only properly manifested containers are accepted at the Detrex facility. The manifest system complies with Rule 299.9608. Careful records are kept of all incoming hazardous wastes in accordance with Rule 299.9609 and biennial reporting is completed in accordance with Rule 299.9610. Section K of this Application provides additional details on manifesting and record keeping.

The following procedures are followed whenever Detrex is receiving and/or transporting hazardous wastes:

1) Detrex transports only properly manifested and labeled shipments of hazardous waste to its solvent recovery operation that are accompanied with appropriate land ban notification as well as other DOT required paperwork and meet the container condition requirements outlined in Section D-1a(1);

2) Only halogenated solvents are accepted at the facility;

3) Shipments are recorded in the customer Hazardous Waste Shipment Ledger upon receipt at the facility;

4) A sample is taken from each container, per the waste analysis plan in Section C, for analysis;

5) The manifest is signed and dated only after approval from the laboratory based on completion of waste screening analysis. One copy of the signed manifest is given immediately to the transporter, if other than Detrex, one copy is returned to the waste generator within 30 days, and one copy is retained at the facility for a period of at least three years;

6) Containers are relabelled using Detrex labelling procedures;

7) Containers are stored in the properly designated, secured area.

D-1a(3) Secondary Containment System Design and Operation
[40 CFR §270.15(a),(b), §264.175(a)]

D-1a(3)(a) Requirement for the Base or Liner to Contain Liquids
[40 CFR §270.15(a)(1), §264.175(b)(1)]

All containerized hazardous wastes are placed in the designated hazardous waste container storage area after being received on site. All process operations and the container storage area are located within a single enclosed building. The building floor, including the container storage and tank storage areas is constructed of concrete with peripheral concrete curbing to provide secondary containment in the reclamation area and the warehouse area. All doorways are diked with concrete ramps to maintain secondary containment while allowing for easy movement of equipment. The container storage area and diked doorways are located on Attachment D-1. Structural drawings, presenting plan views, elevations, sections, supplementary views, and specifications on construction materials for the Eaton Avenue facility are presented in Attachment D-2.

The peripheral concrete curbing is coated with Surewall Surface Bonding Cement Coating. There are no floor drains or other such openings in the secondary containment area. All existing expansion joints and any cracks will be cleaned out, saw cut if necessary to provide a clean opening. Subsequently all expansion joints and any cracks will be filled with a compatible chemically resistant flexjoint sealant. The selected sealant will be installed in accordance with the manufacturer's specifications. The concrete slab and perimeter diking, with sealed joints, will serve as an impervious containment structure.

D-1a(3)(b) Containment System Drainage
[40 CFR §270.15(a)(2), §264.175(b)(2)]

The containers within the container storage areas are placed on wooden pallets to elevate them off the concrete floor. Containers are also provided with adequate aisle space to allow for routine inspection. If an inspector discovers the presence of liquid, clean-up will be conducted immediately. If necessary, the forklift can be used to move containers in order to identify the leaking container(s) and to complete the necessary clean-up.

D-1a(3)(c) Containment System Capacity
[40 CFR §270.15(a)(3), §264.175(b)(3)]

The total capacity of the secondary containment system at the Detrex facility is approximately 25,301 gallons (see Attachment D-4 for calculation and certification). The maximum volume of hazardous waste stored at the facility is 13,750 gallons. Thus, even under maximum inventory conditions, secondary containment is far greater than the inventory.

D-1a(3)(d) Control of Run-On [40 CFR §270.15(a)(4), §264.175(b)(4)]

Run-on into the containment system is eliminated by the building structure. All precipitation is controlled outside of the facility and drainage is promoted away from the building.

D-1a(4) Removal of Liquids from Containment System
[40 CFR §270.15(a)(5), §264.175(b)(5)]

Routine inspections for the presence of spilled or leaked materials within the secondary containment system are conducted. The inspection items and schedule are presented in Section F of this operating license application. As required, accumulated liquids are immediately collected from the secondary containment system.

Clean-up procedures, which may include notification of a spill clean-up firm, furnishing the clean-up crew with physical and/or chemical properties of waste and amount of waste released, shall be implemented.

a) Clean-up of released waste: The waste will be collected via use of absorbent material for small spills. The collected material will then be placed in a DOT-approved container(s) and stored in a segregated area of the container storage area. The container(s) will be clearly labelled describing the source of the material. The material will subsequently be characterized as F001 or F002 material based on the source of the spilled material. The material would then be transported to an off-site facility permitted to receive the material in accordance with all state and federal regulations. For large spills, the waste will be collected with a wet-vac or pump and placed into DOT-approved containers in the container storage area. The waste would be characterized based on the source of the spilled material and subsequently be reclaimed (recycled)

on site. Residual material would be handled as described above for small spills.

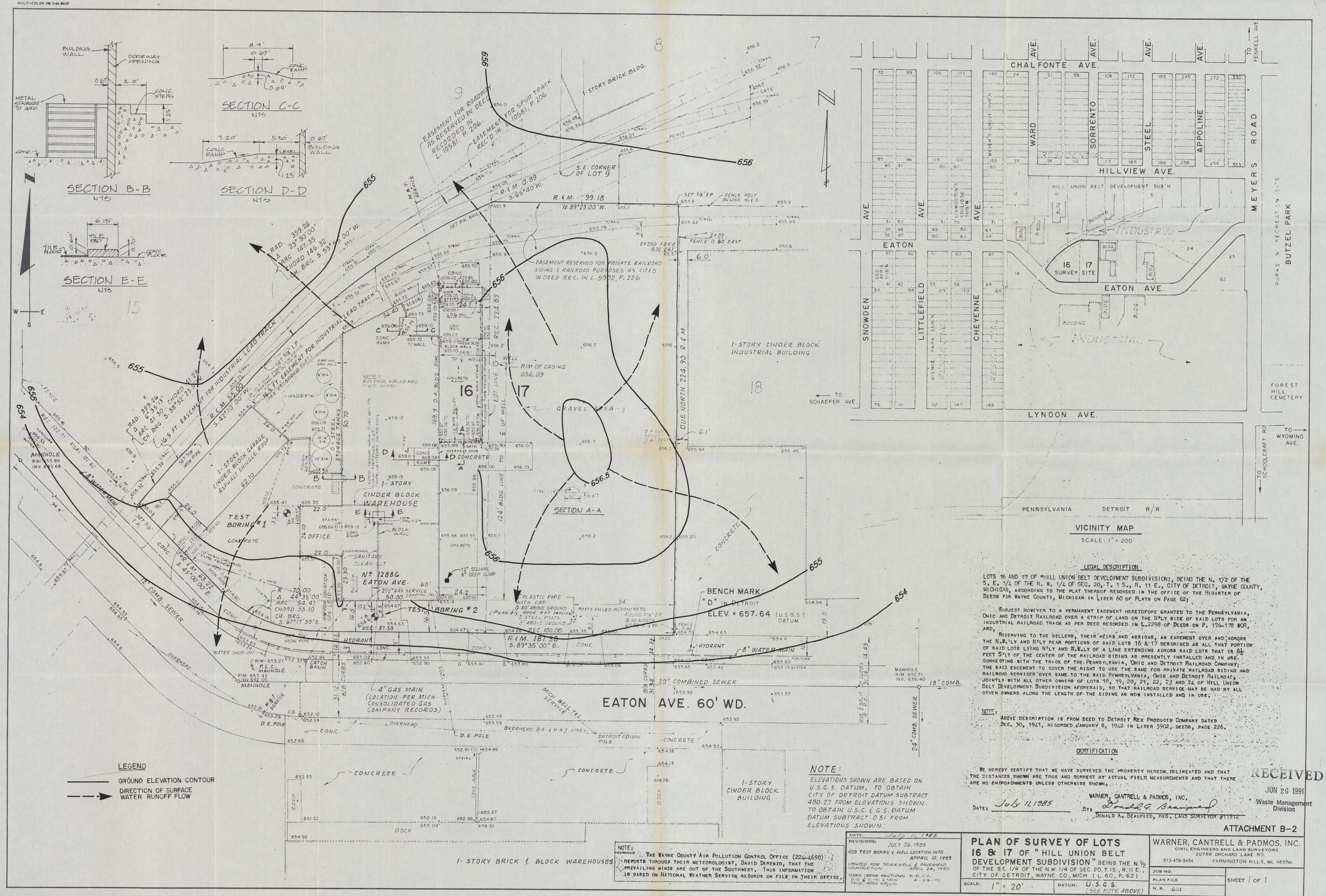
b) Decontamination: Following clean-up with absorbent material, the affected area of the secondary containment area will be swept and all sweepings will be containerized and handled as identified above for small spills. The pad may be subsequently decontaminated by steam cleaning. Any wash waters generated will be collected in DOT-approved containers and transported off site for treatment/disposal at a permitted facility as described above unless the material meets the specifications for discharge in accordance with the facility's Detroit Water and Sewerage Department Wastewater Discharge Permit.

potentially leaking material toward the perimeter of the tank where visual inspection would detect potential leakage.

D-2d Tank Operation [40 CFR §264.194]

With the recent redesignation of the two former process tanks as hazardous waste storage tanks, Detrex intends to operate the two tanks at less than their nominal capacity. The total tank storage design capacity of the Eaton Avenue facility is 5,500 gallons. The total nominal capacity of the two tanks is 6,800 gallons. Tank 12 will be operated at a storage capacity of 1,900 gallons and Tank 22 will be operated at a storage capacity of 3,600 gallons.

A permanent painted marking has been placed on each tank and on the sight glass which provides level control. By visually ensuring the level in either tank does not rise above the marked level, Detrex will ensure the design capacity is not exceeded. The sight glass is inspected daily, at minimum, as well as at any time that material is being charged into the tanks.



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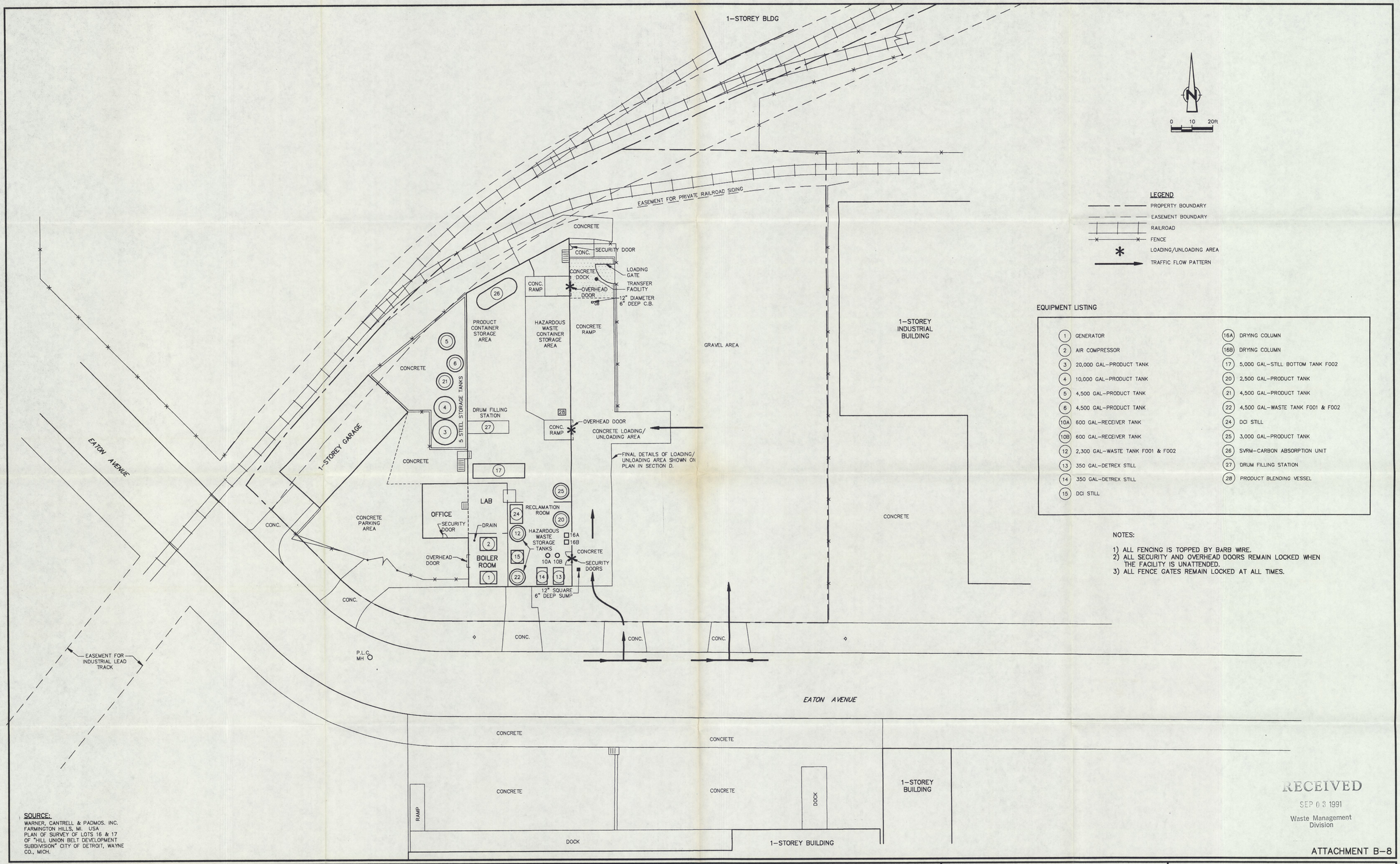
Waste Management Division

ATTACHMENT B-2



№	Revision	Date	Initial

Approved

DETREX CORPORATION
EATON AVENUE FACILITY, DETROIT

PERMIT APPLICATION

ACCESS CONTROL

CRA
CONESTOGA-ROVERS & ASSOCIATES

Drawn by: R.B.B.	Scale: 1" = 20'	Date: JUNE 1991	File №: 58 (P-07)	Rev.№: –
Designed by: B.McC.				
Checked by: E.R.	Field book:	Project №: 2471	Drawing №:	

KEY TO MAP

500-Year Flood Boundary	
100-Year Flood Boundary	
Zone Designations* With Date of Identification e.g., 12/2/74	ZONE B / ZONE B
100-Year Flood Boundary	
500-Year Flood Boundary	
Base Flood Elevation Line With Elevation In Feet**	513
Base Flood Elevation In Feet Where Uniform Within Zone**	(EL 987)
Elevation Reference Mark	RM7×
River Mile	•M1.5

**Referenced to the National Geodetic Vertical Datum of 1929

*EXPLANATION OF ZONE DESIGNATIONS

ZONE	EXPLANATION
A	Areas of 100-year flood; base flood elevations and flood hazard factors not determined.
A0	Areas of 100-year shallow flooding where depths are between one (1) and three (3) feet; average depths of inundation are shown, but no flood hazard factors are determined.
AH	Areas of 100-year shallow flooding where depths are between one (1) and three (3) feet; base flood elevations are shown, but no flood hazard factors are determined.
A1-A30	Areas of 100-year flood; base flood elevations and flood hazard factors determined.
A99	Areas of 100-year flood to be protected by flood protection system under construction; base flood elevations and flood hazard factors not determined.
B	Areas between limits of the 100-year flood and 500-year flood; or certain areas subject to 100-year flooding with average depths less than one (1) foot or where the contributing drainage area is less than one square mile; or areas protected by levees from the base flood. (Medium shading)
C	Areas of minimal flooding. (No shading)
D	Areas of undetermined, but possible, flood hazards.
V	Areas of 100-year coastal flood with velocity (wave action); base flood elevations and flood hazard factors not determined.
V1-V30	Areas of 100-year coastal flood with velocity (wave action); base flood elevations and flood hazard factors determined.

NOTES TO USER

Certain areas not in the special flood hazard areas (zones A and V) may be protected by flood control structures.

This map is for flood insurance purposes only; it does not necessarily show all areas subject to flooding in the community or all planimetric features outside special flood hazard areas.

For adjoining map panels, see separately printed Index To Map Panels.

INITIAL IDENTIFICATION:
JULY 26, 1974

FLOOD HAZARD BOUNDARY MAP REVISIONS:
FEBRUARY 7, 1975

FLOOD INSURANCE RATE MAP EFFECTIVE:
JULY 2, 1981

FLOOD INSURANCE RATE MAP REVISIONS:

Refer to the FLOOD INSURANCE RATE MAP EFFECTIVE date shown on this map to determine when actuarial rates apply to structures in the zones where elevations or depths have been established.

To determine if flood insurance is available in this community, contact your insurance agent, or call the National Flood Insurance Program, at (800) 638-6620.

APPROXIMATE SCALE
1000 0 1000 FEET

NATIONAL FLOOD INSURANCE PROGRAM

FIRM
FLOOD INSURANCE RATE MAP

CITY OF
DETROIT,
MICHIGAN
WAYNE COUNTY

PANEL 5 OF 45
(SEE MAP INDEX FOR PANELS NOT PRINTED)

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COMMUNITY-PANEL NUMBER
260222 0005 B

EFFECTIVE DATE:
JULY 2, 1981

federal emergency management agency
federal insurance administration

DETREX CORPORATION

FEDERAL INSURANCE ADMINISTRATION
(FIA) FLOOD MAP

ATTACHMENT B–10

C.R.A. PROJECT No.2471 DWG. No. 04 FILE: S