OCT 2 6 1984
Rev. 0
A - 1

ASHLAND CHEMICAL COMPANY

RESEARCH & DEVELOPMENT LABORATORY

5200 BLAZER PARKWAY

DUBLIN, OH 43017

EPA IDENTIFICATION NUMBER OHD042311209

Generator
Transporter
Storage

Please print or type in the unshaded areas only (fill-in areas are spaced for elite type, i.e., 12 characters/inch).

Ashland Chemical Company - Dublin, OH R&D OCT 2 6 1984

Form Approved OMB No. 158-R0175

FORM 1 GENERAL

EPA

U.S. ENVIRONMENTAL PROTECTION AGENCY

GENERAL INFORMATION

Consolidated Permits Program

(Read the "General Instructions" before starting.)

I. EPA I.D. NUMBER

F O H D 0 4 2 3 1 1 2 0 9 D

Rev 1 A-2

GENERAL INSTRUCTIONS

If a preprinted label has been provided, affix it in the designated space. Review the information carefully; if any of it is incorrect, cross through it and enter the correct data in the appropriate fill-in area below. Also, if any of the preprinted data is absent *(the area to the left of the label space lists the information that should appear)*, please provide it in the proper fill-in area(s) below. If the label is complete and correct, you need not complete items I, III, V, and VI *(except VI-B which must be completed regardless)*. Complete all items if no label has been provided. Refer to the instructions for detailed item descriptions and for the legal authorizations under which this data is collected.

LABEL ITEMS

I. I.D. NUMBER

III. FACILITY NAME

V. FACILITY MAILING ADDRESS

VI. FACILITY LOCATION

JAN 1 1 1985 REV 1 A-2

PLEASE PLACE LABEL IN THIS SPACE

II. POLLUTANT CHARACTERISTICS

INSTRUCTIONS: Complete A through J to determine whether you need to submit any permit application forms to the EPA. If you answer "yes" to any questions, you must submit this form and the supplemental form listed in the parenthesis following the question. Mark "X" in the box in the third column if the supplemental form is attached. If you answer "no" to each question, you need not submit any of these forms. You may answer "no" if your activity is excluded from permit requirements; see Section C of the instructions. See also, Section D of the instructions for definitions of bold-faced terms.

SPECIFIC QUESTIONS	MARK 'X' YES	NO	FORM ATTACHED
A. Is this facility a publicly owned treatment works which results in a discharge to waters of the U.S.? (FORM 2A)		X	
C. Is this a facility which currently results in discharges to waters of the U.S. other than those described in A or B above? (FORM 2C)		X	
E. Does or will this facility treat, store, or dispose of hazardous wastes? (FORM 3)	X		X
G. Do you or will you inject at this facility any produced water or other fluids which are brought to the surface in connection with conventional oil or natural gas production, inject fluids used for enhanced recovery of oil or natural gas, or inject fluids for storage of liquid hydrocarbons? (FORM 4)		X	
I. Is this facility a proposed stationary source which is one of the 28 industrial categories listed in the instructions and which will potentially emit 100 tons per year of any air pollutant regulated under the Clean Air Act and may affect or be located in an attainment area? (FORM 5)		X	

SPECIFIC QUESTIONS	MARK 'X' YES	NO	FORM ATTACHED
B. Does or will this facility *(either existing or proposed)* include a concentrated animal feeding operation or aquatic animal production facility which results in a discharge to waters of the U.S.? (FORM 2B)		X	
D. Is this a proposed facility *(other than those described in A or B above)* which will result in a discharge to waters of the U.S.? (FORM 2D)		X	
F. Do you or will you inject at this facility industrial or municipal effluent below the lowermost stratum containing, within one quarter mile of the well bore, underground sources of drinking water? (FORM 4)		X	
H. Do you or will you inject at this facility fluids for special processes such as mining of sulfur by the Frasch process, solution mining of minerals, in situ combustion of fossil fuel, or recovery of geothermal energy? (FORM 4)		X	
J. Is this facility a proposed stationary source which is NOT one of the 28 industrial categories listed in the instructions and which will potentially emit 250 tons per year of any air pollutant regulated under the Clean Air Act and may affect or be located in an attainment area? (FORM 5)		X	

III. NAME OF FACILITY

1 SKIP A S H L A N D C H E M I C A L C O M P A N Y

IV. FACILITY CONTACT

A. NAME & TITLE *(last, first, & title)*: 2 H E N D R I C K S O N A R L E N E A E N V R E N G R

B. PHONE *(area code & no.)*: 6 1 4 8 8 9 3 6 9 5

V. FACILITY MAILING ADDRESS

A. STREET OR P.O. BOX: 3 P O B O X 2 2 1 9

B. CITY OR TOWN: 4 C O L U M B U S

C. STATE: O H

D. ZIP CODE: 4 3 2 1 6

VI. FACILITY LOCATION

A. STREET, ROUTE NO. OR OTHER SPECIFIC IDENTIFIER: 5 5 2 0 0 B L A Z E R P A R K W A Y

B. COUNTY NAME: F R A N K L I N

C. CITY OR TOWN: 6 D U B L I N

D. STATE: O H

E. ZIP CODE: 4 3 0 1 7

F. COUNTY CODE *(if known)*:

CONTINUE ON REVERSE

CONTINUED FROM THE FRONT

FEB 07 1985

VII. SIC CODES (4-digit, in order of priority)

A. FIRST	B. SECOND
7999 (specify) Non-classifiable Establishments	(specify)
C. THIRD	**D. FOURTH**
(specify)	(specify)

Rev. 1

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VIII. OPERATOR INFORMATION

A. NAME: ASHLAND CHEMICAL COMPANY

B. Is the name listed in Item VIII-A also the owner? ☐ 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 614 889 3333

E. STREET OR P.O. BOX: 5200 BLAZER PARKWAY

F. CITY OR TOWN: DUBLIN

G. STATE: OH

H. ZIP CODE: 43017

IX. INDIAN LAND

Is the facility located on Indian lands? ☐ YES ☒ NO

X. EXISTING ENVIRONMENTAL PERMITS

A. NPDES (Discharges to Surface Water): N	**D. PSD** (Air Emissions from Proposed Sources): 9 P
B. UIC (Underground Injection of Fluids): U	**E. OTHER** (specify): 9 (specify)
C. RCRA (Hazardous Wastes): 9	**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 boundaries. 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)

Research and Development Lab for Ashland Chemical Company

XIII. CERTIFICATION (see instructions)

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
Michael E. Mullier - Research Manager James D. Idol, Jr.- Vice President Venture Research & Development		Nov 12/84 Nov 20/84

COMMENTS FOR OFFICIAL USE ONLY

(fill—in areas are spaced for elite type, i.e., 12 characters/inch).

Form Approved OMB No. 158-S80004

FORM 3 RCRA

EPA

U.S. ENVIRONMENTAL PROTECTION AGENCY

HAZARDOUS WASTE PERMIT APPLICATION

Consolidated Permits Program

(This information is required under Section 3005 of RCRA.)

I. EPA I.D. NUMBER

F O H D 0 4 2 3 1 1 2 0 9 — 1

FOR OFFICIAL USE ONLY

APPLICATION RECEIVED	DATE RECEIVED (yr., mo., & day)	COMMENTS
		FEB 10 1986 Rev. A-4

II. FIRST OR REVISED APPLICATION

Place 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 or a revised 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's EPA I.D. Number in Item I above.

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

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

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

C 8 — YR. | MO. | DAY

☐ 2. NEW FACILITY *(Complete item below.)*

FOR NEW FACILITIES, PROVIDE THE DATE *(yr., mo., & day)* OPERATION BEGAN OR IS EXPECTED TO BEGIN

YR. | MO. | DAY

B. REVISED APPLICATION *(place an "X" below and complete Item I above)*

☒ 1. FACILITY HAS INTERIM STATUS

☐ 2. FACILITY HAS A RCRA PERMIT

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 for 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, then 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
LAND APPLICATION	D81	ACRES OR HECTARES
OCEAN 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 OF MEASURE 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 1

LINE NUMBER	A. PROCESS CODE (from list above)	B. PROCESS DESIGN CAPACITY 1. AMOUNT (specify)	B. 2. UNIT OF MEASURE (enter code)	FOR OFFICIAL USE ONLY	LINE NUMBER	A. PROCESS CODE (from list above)	B. PROCESS DESIGN CAPACITY 1. AMOUNT	B. 2. UNIT OF MEASURE (enter code)	FOR OFFICIAL USE ONLY
X-1	S 0 2	600	G		5				
X-2	T 0 3	20	E		6				
1	S 0 1	22,000	G		7				
2	S 0 2	8,000	G		8				
3					9				
4					10				

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Continued from page 2.
NOTE: Photocopy this page before completing if you have more than 26 wastes to list.

Form Approved OMB No. 158-S80004

EPA I.D. NUMBER (enter from page 1)	FOR OFFICIAL USE ONLY	
W O H D 0 4 2 3 1 1 2 0 9 — T/A C 1	W DUP — T/A C 2 DUP	A-6

DESCRIPTION OF HAZARDOUS WASTES (continued)

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))
1	F002	4	T	S01	
2	F003	22	T	S01	
3	F005				Included with above.
4	D001	201	T	S01	
5	F001	2	T	S01	
6	F002	2	T	S01	
7	D002	3	T	S01	
8	D001	25	T	S02	
9	F001				Included with above.
10	F002				Included with above.
11	F003				Included with above.
12	F005				Included with above.
13	F003	1	T	S01	
14	F005	1	T	S01	
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					

EPA Form 3510-3 (6-80)

CONTINUE ON REVERSE

(enter "A", "B", "C", etc. behind the "3" to identify photocopied pages)

Continued from the front.

IV. DESCRIPTION OF HAZARDOUS WASTES (continued)

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

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A-7

EPA I.D. NO. (enter from page 1)														
S	F	O	H	D	0	4	2	3	1	1	2	0	9	T/A C 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)
40 05 026	083 08 007

VIII. FACILITY OWNER

☐ A. If the facility owner is also the facility operator as listed in Section VIII on Form 1, "General Information", place an "X" in the box to the left and skip to Section IX below.

B. If the facility owner is not the facility operator as listed in Section VIII on Form 1, complete the following items:

1. NAME OF FACILITY'S LEGAL OWNER	2. PHONE NO. (area code & no.)
E Rock Hill Associates, c/o Shearson Lehman/American Express, Inc.	212-321-6000

3. STREET OR P.O. BOX	4. CITY OR TOWN	5. ST.	6. ZIP CODE
F American Express Plaza, 125 Broad St.	G New York	NY	10004

IX. OWNER CERTIFICATION

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this and all attached documents, and that based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted 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 (print or type)	B. SIGNATURE	C. DATE SIGNED
William M. Kahn General Partner	William M Kahn	3/24/86

X. OPERATOR CERTIFICATION

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this and all attached documents, and that based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted 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 (print or type)	B. SIGNATURE	C. DATE SIGNED
James D. Idol, Jr.	James D. Idol Jr.	3/3/86

CONTINUE ON PAGE 5

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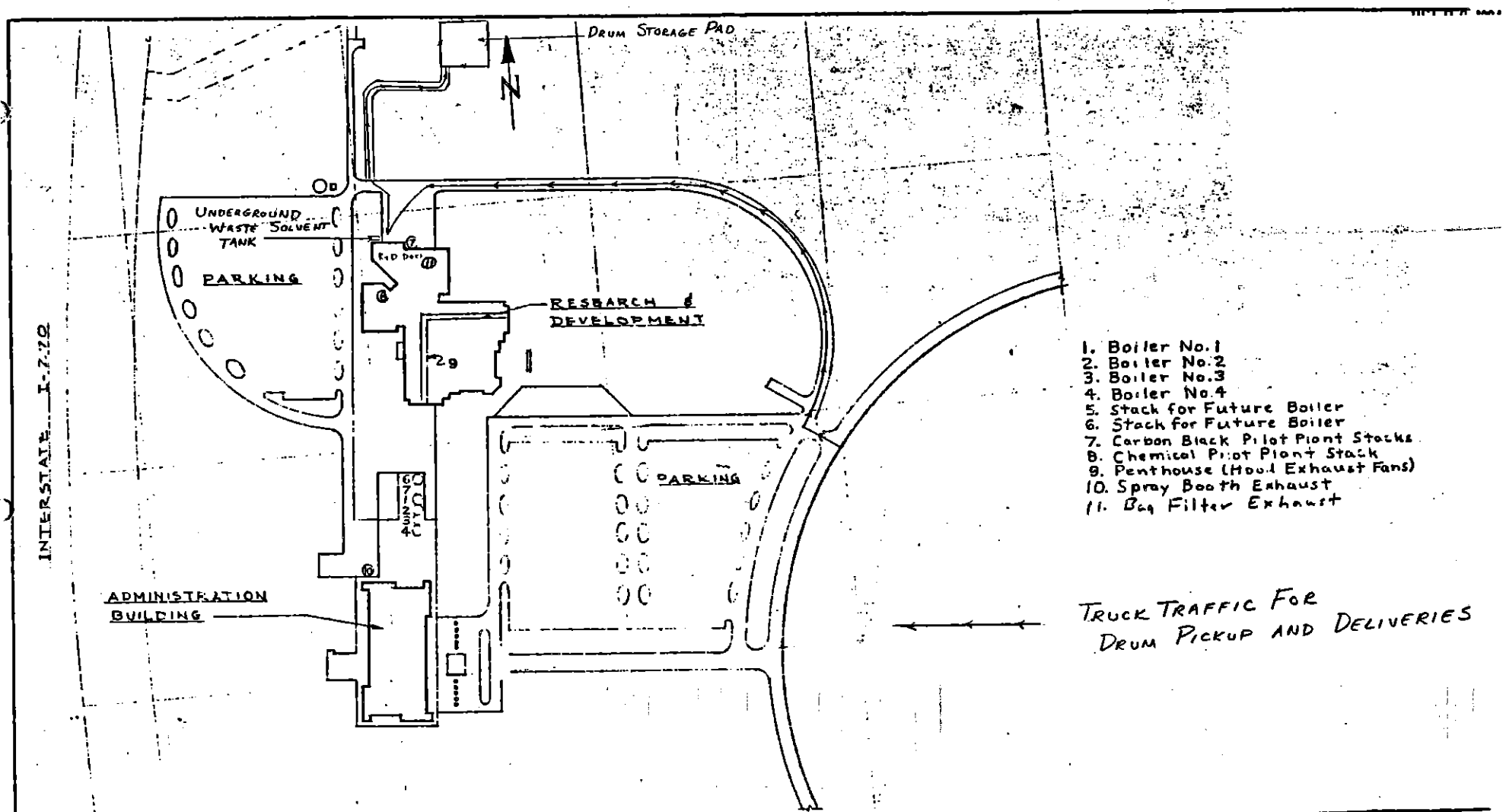

REV.	DATE	DESCRIPTION	APPR.

Ashland ASHLAND CHEMICAL CO.

Emission Sources Plot Plan

SCALE | DR.BY
DATE | CK.BY

FEB 19 1986

Rev. 1
A-8

HILLIARD, OHIO
7 1/2'

N

NORTHWEST COLUMBUS
OHIO
7 1/2'

OCT 26 1984

Rev. 6

HILLIARD QUADRANGLE
OHIO
7.5 MINUTE SERIES (TOPOGRAPHIC)

SW/4 DUBLIN 15' QUADRANGLE

UNITED STATES
DEPARTMENT OF THE I
GEOLOGICAL SURVE

OCT 26 1984
Rev. C
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HAZARDOUS WASTE FACILITY PERMIT APPLICATION

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SECTION B
FACILITY DESCRIPTION

B-1 General Description

Ashland Chemical Research and Development Laboratory is located in the Village of Dublin which is northwest of the City of Columbus, OH. The R&D Laboratory is next to the administrative headquarters building of Ashland Chemical Company.

The R&D Laboratory location is shown on the Hilliard, Ohio USGS topographic map which is attached to Part A of this application. The lab is also shown on the topographic map Map No. G-8 in the Appendix.

The street address of the R&D Laboratory is:

Ashland Chemical Company
Research & Development Laboratory
5200 Blazer Parkway
Dublin, OH 43017

The mailing address is:

Ashland Chemical Company
Research & Development Laboratory
P.O. Box 2219
Columbus, OH 43216

The Ashland R&D Laboratory is the main research and development facility for Ashland Chemical. In this capacity Ashland is involved in research in carbon black, foundry products, polyester resins, specialty polymers, adhesives, electronic and laboratory chemicals, petrochemicals, industrial chemicals and solvents and polymers. In addition to these business areas in which we are currently operating, Ashland Chemical does research in areas which are not related to our current lines of business.

Research is done in the laboratory as well as on the pilot plant scale. During this research various chemicals are made and numerous by-products are made which must be ultimately sent off site for disposal. The majority of these wastes are stored in 55 gallon drums on a drum storage pad north of the R&D Laboratory.

In addition to these drummed wastes, wash solvent from the cleaning of reactors in the pilot plant and line flush solvents are stored in an 8000 gallon underground tank beside the R&D laboratory on the north side. These wash solvents and line flush solvents will be incinerated. The tank has been added to the Part A application included in this Part B and will be further addressed in this Part B. The normal operation of this tank may involve sampling of the contents where it is nearly full to determine its final disposition. If the analysis of the contents indicates that it cannot be incinerated, Ashland will look for alternate means of disposal. In most cases it is possible to characterize the waste based on knowledge of the solvents used to clean the equipment and flush lines and the resin residue on the equipment.

The hazardous wastes which Ashland seeks to store are listed in Part A of this application.

The contact for any questions regarding hazardous waste management at Ashland Chemical in Dublin, Ohio is:

Arlene A. Hendrickson
Environmental Engineer
614/889-3695

B-2 Topographic Map

A topographic map of the R&D Laboratory is in the appendix of this permit application. The topographic map is in 6 Sections: Map Nos. F-7, F-8, G-7, G-8, H-7 and H-8. The scale of the maps is 1 inch equals 100 feet. The maps show 2 foot contour intervals of elevation. It was necessary to include all 6 maps in order to show a distance of 1000 feet around the entire R&D lab and Ashland Chemical Administrative Building property. Legal boundaries of Ashland Chemical property are indicated. The map north arrow is in the lower left border on the map. The only surface waters within 1000 feet are the Cosgray Ditch to the north, George Geary Ditch to the west and southwest, and Cramer Ditch to the southwest. As indicated on the topographic maps, these are all creeks with intermittant flow.

The R&D Laboratory and drum storage pad is shown in the upper left hand corner of Map No. G-8.

R&D Laboratory Layout - The Dublin complex consists of the administration Building and parking lot located to the south which is indicated on Map Nos. G-8 and G-7. The R&D laboratory is located directly north of the Administration Building and has a parking lot to the west of the lab.

The hazardous waste storage area is located on the drum storage pad north of the R&D Laboratory. Hazardous wastes have been stored in this area in accordance with interim status standards for waste containers (40 CFR Part 265 Subpart I, Use And Management Of Containers). This storage area will be upgraded to meet the final standards for waste containers as specified in 40 CFR Part 264 Subpart I. The storage area will be constructed to the specifications shown in Section D.

All transfer of leaking drums will be done in the waste storage area of the drum storage pad.

The underground waste storage tank is located north of the R&D Laboratory west of the R&D Dock. Hazardous wastes have been stored in the tank in accordance with interim status standards for tanks (40 CFR Part 265 Subpart J, Tanks).

Site Drainage & Run-Off Control - As shown on the topographic map the R&D Laboratory area is essentially flat. Surface drainage is normally limited to runoff from a heavy rainfall. All catch basins on the property drain to Cosgray Ditch north of the R&D building.

Inadvertent spills in the waste drum storage area will be confined to the storage pad with the construction of the ramp and curbing. Inadvertent spills in the area near the underground tank where drums of spent wash solvents are pumped into the tank will be confined to the solvent storage room. An ample supply of shovels, rakes, absorbent pads, sand and dirt is available to encircle a spill. Bags of absorbent materials are stocked in the R&D basement for absorbing spills. The hazardous waste storage area is inspected weekly for spills, leaks or damaged or deteriorating drums. Empty drums are available into which the contents of a damaged or deteriorating drum can be placed. The waste storage tank is inspected weekly by gauging the tank. The level in the tank is also measured prior to pumping any additional solvents into the tank to determine if there is a significant change in the amount of solvents in the tank. The fill pipe is inspected each time material is pumped into the tank.

Legal Boundaries and Access Control - The plant legal boundaries are shown on the topographic maps indicated by PL. the land upon which the Administration Building and R&D Laboratory is situated and the surrounding area indicated within the property line is owned by Ashland Oil, Inc. Ashland Chemical Company is a division of Ashland Oil, Inc. Ashland Oil, Inc. leases the parcels of land upon which the buildings are located to Rock Hill Associates. On October 16, 1984 Rock Hill Associates, c/o Shearson/American Express purchased the Administration Building and R&D Laboratory.

The entire drum storage area is enclosed by a 6' 9½" high chainlink fence. Access to the drum storage area is through two gates:

1. The south gate which is the main gate used
2. The northeast gate which is used only for access to the well pump

The south gate is kept open only during normal working hours approximately 7:00 a.m. to 5:30 p.m. It is locked shut on weekends and other times when the lab is not operating. The northeast gate is kept locked at all times unless work is being done on the pump.

The fill pipe to the underground tank is located in the solvent storage room of the R&D Building. The R&D Building is open during normal working hours and locked during non working hours except for the northwest door which has a guard in constant attendance. In addition to this guard there is a TV monitor on the R&D Dock which is the closest entrance to the solvent storage room. This TV monitor is watched by the guard on the main reception desk in the Administration Building as well as the guard in the R&D Building.

Surrounding Land Use - The R&D Laboratory is located in an area zoned Office Lab Research. The area south of Rings road, which is south of the Ashland property, is zoned residential. The area to the west of the Ashland property on the other side of Interstate 270 is also zoned residential. The Metro Center which is north of Ashland Chemical is zoned Planned Unit Development and Office.

Injection Wells & Withdrawal Wells - There are no injection wells on the Ashland site or 1,000 feet surrounding the site.

The water well which supplies water to the Ashland facility is located at the northeast corner of the drum storage pad as shown on the drawing in the Appendix.

Wind Rose - A wind rose for the Columbus Airport is attached.

B-3 Location Information

Seismic Standard - Since this is not a new facility, the seismic standard does not apply.

Floodplain - The R&D Laboratory is located in an area outside the 500 year floodplain. On the Flood Insurance Rate Map Community Panel Number 390673 0005 B of the Village of Dublin, Ohio, Franklin and Delaware Counties, the R&D Laboratory and the drum storage pad are located in Zone C. Zone C is an area outside the 500 year flood. Therefore, the facility is not in the 100 year flood plain.

Traffic Pattern - The traffic pattern at the R&D Laboratory is sketched on the attached site plan.

On-Site Traffic Patterns - The road surface of the driveways at the R&D Laboratory is asphalt. The load bearing capabilities have been sufficient over the years for delivery trucks fully loaded with product. There is no reason to believe that the roads will not be adequate for trucks fully loaded with wastes since the wastes will have specific gravities similar to the raw materials delivered. Approximate gross weight of the largest truck is:

	Pounds
Tractor Unit	16,500
42' Van	12,600
Truckload (80 55-gallon drums)	33,000
Total	62,100

The truck traffic would be approximately 25 trucks per week which would use the same route to make deliveries to either the R&D dock or Administration Building dock. The volume of car traffic would be approximately 150 per day which would be the R&D employees parking in the lot behind the R&D Laboratory.

Off-Site Traffic Patterns

1) Trucks leaving the Ashland Chemical buildings would turn left onto Blazer Parkway.

2) Blazer Parkway deadends at Frantz Road. The trucks would then turn left onto Frantz Road.

3) At the intersection of Frantz Road at U.S. 33 (S.T. 161) the trucks would turn left.

4) The truck could then immediately get on I-270 north or I-270 south.

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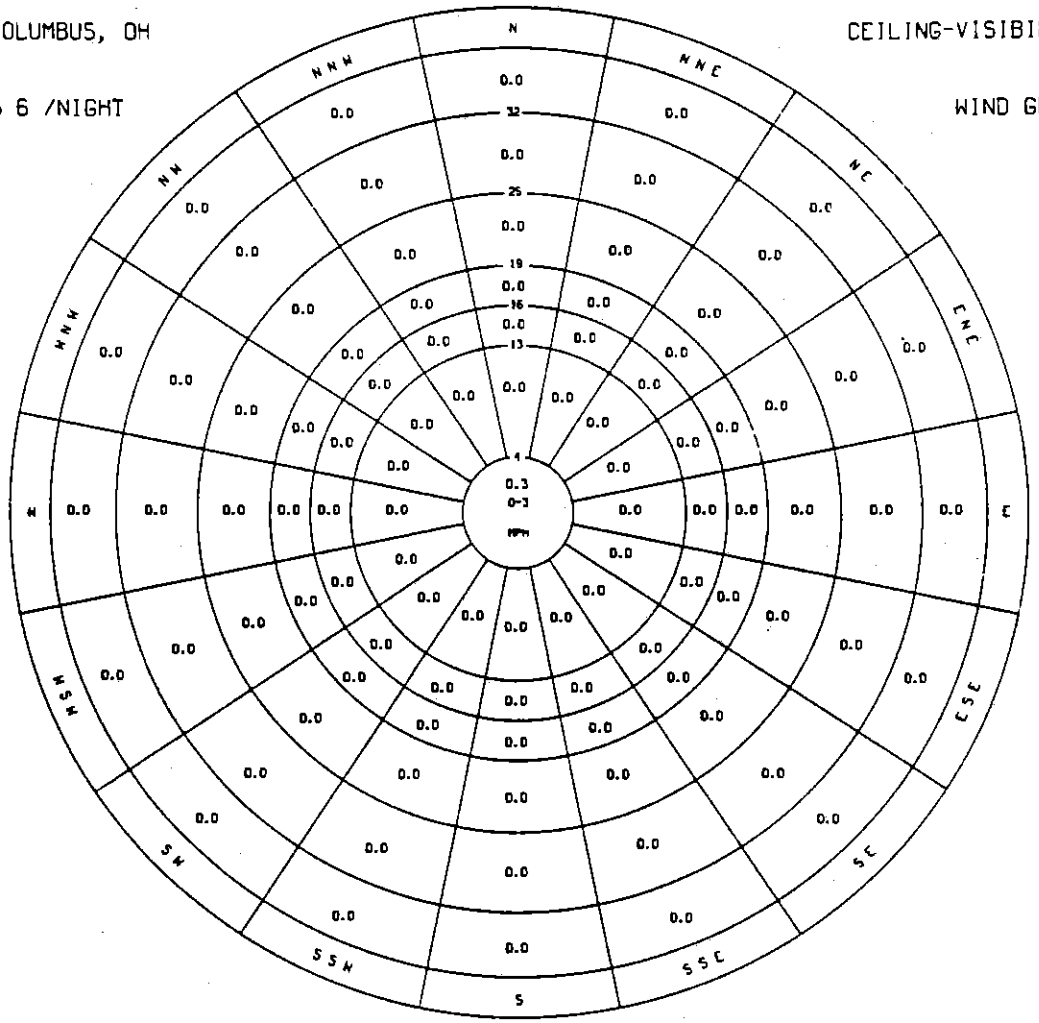
CMH COLUMBUS, OH

CLASS 6 /NIGHT

CEILING-VISIBILITY

WIND GRAPH



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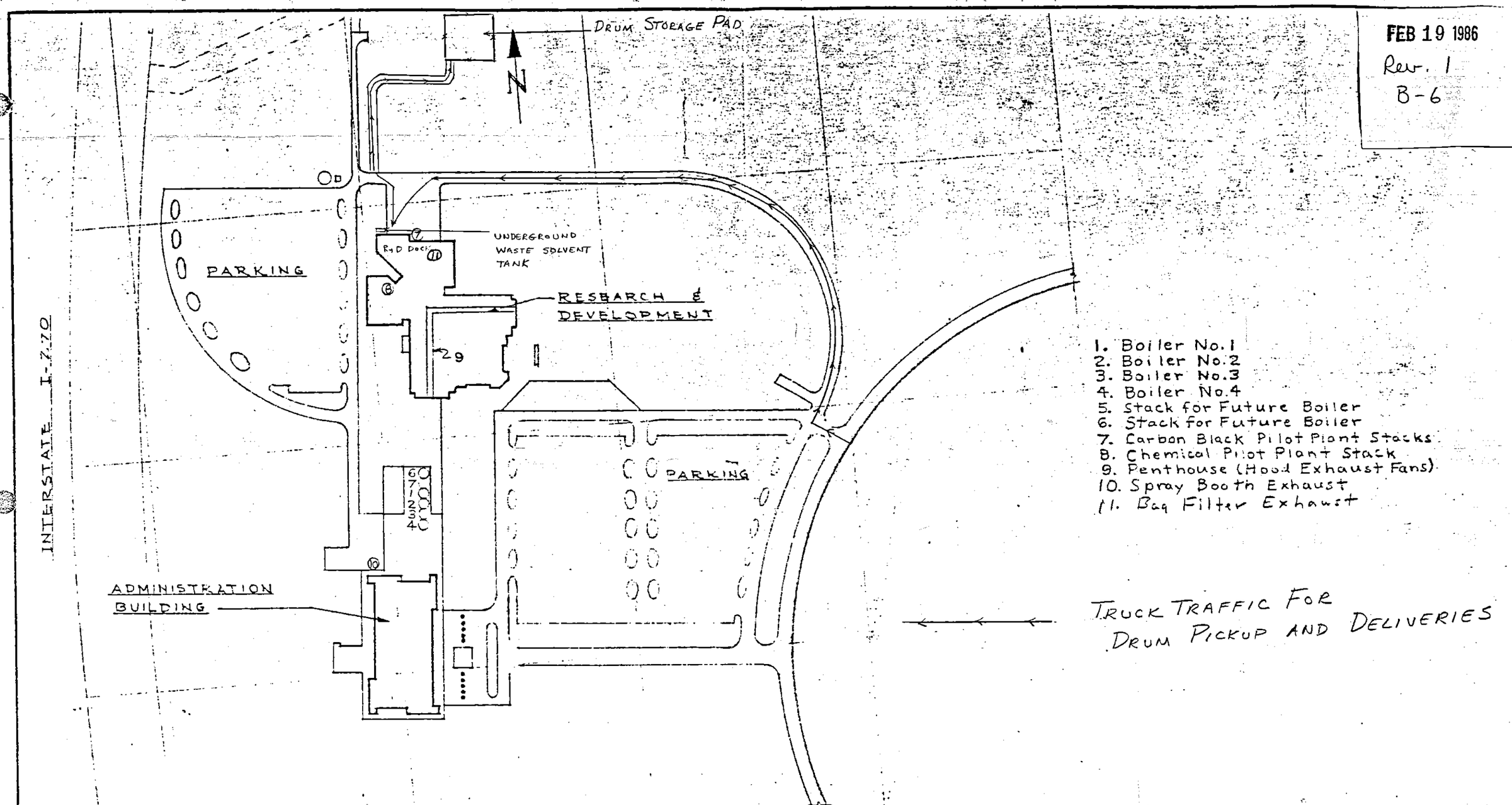

1. Boiler No. 1
2. Boiler No. 2
3. Boiler No. 3
4. Boiler No. 4
5. Stack for Future Boiler
6. Stack for Future Boiler
7. Carbon Black Pilot Plant Stacks
8. Chemical Pilot Plant Stack
9. Penthouse (Hood Exhaust Fans)
10. Spray Booth Exhaust
11. Bag Filter Exhaust

REV.	DATE	DESCRIPTION	APPR.

Ashland ASHLAND CHEMICAL CO.

Emission Sources Plot Plan

SCALE	DR. BY
DATE	CK. BY

SECTION C

WASTE CHARACTERISTICS

The Resource Conservation and Recovery Act, Part 264 (final standards) and Part 265 (interim status standards) require owners/operators of a hazardous waste treatment, storage and disposal facility to have a written waste analysis plan.

Ashland Chemical, Inc. facilities that store laboratory generated waste longer than 90 days are required to prepare a Waste Analysis Plan. The plan must satisfy the following regulatory requirements for a storage facility:

1. Before waste is stored a detailed physical and chemical analysis must be obtained from a representative sample 122.25(a)(2) and 264.13(a)(1).

2. The analysis may include data developed under Part 261 of RCRA and existing published or documented data on the hazardous waste 264.13(a)(2).

3. The analysis must be repeated frequently enough to insure the analysis is accurate and up-to-date 264.13(a)(3) and (b)(4).

4. The written plan must specify parameters identified and rational employed in the selection of the parameters 122.25(a)(3) and 264.13(b)(1).

5. The test methods which are used to obtain the parameters must also be identified. The selected test methods must be sufficient to insure compliance with Section 264.17 264.13(b)(2) and (b)(6).

6. The sampling method employed for collection of a representative sample must be specified for each waste 264.13(b)(3).

In addition, Ashland Chemical, Inc. must comply with the provisions of 40 CFR 268 (Land Disposal Restrictions) if the wastes generated contain chemical constituents subject to these restrictions. Specifically, the waste analysis and recordkeeping requirements of 40 CFR 268.7 are adhered to. No disposal activities take place at the facility. All wastes generated are shipped off-site to permitted TSD facilities and the appropriate notifications and certifications are made to the TSD. The recordkeeping requirements outlined in 40 CFR 268.7 are also followed.

C-1 CHEMICAL AND PHYSICAL ANALYSES [122.25(a)(2) and 264.13(a)(1) and (a)(2)]

The EPA hazardous wastes are identified on the Notification and Part A forms. The lab generates and stores four categories of hazardous waste:

1. Spent organic solvents mixture with/without organic resins and monomers from research labs.

2. Waste acids.

3. Filter cake contaminated with organic solvents.

4. Spent reactor wash solvents and line flush solvents.

The composition of the solvent mixture (Item 1) is known based upon knowledge of the solvents used in the particular lab. An internal chemical waste profile sheet is completed for each drum of waste generated in the labs. The internal chemical waste profile sheet lists the components of each drum. Physical and chemical properties of each component are known. In most cases mixed solvents are classified as D001 because the majority of the individual solvents flash under 140°F.

Waste acids (Item 2) can be similarly classified based upon the activities in the labs which are using acids. These would be classified as D002 corrosive wastes.

Filter cake contaminated with organic solvents (Item 3) can also be classified based on the material being filtered. Most of these would be classified D001 from filtering ignitable solvents.

Spent reactor wash solvents and line flush solvents (Item 4) can be classified based upon knowledge of the wash solvent being used and residue remaining in the reactors from resin production and solvents in the lines. These wastes would be classified as D001, F001, F002, F003 or F005 depending on the solvents used to clean the reactors or flush the lines.

All of these wastes may contain constituents subject to TCLP requirements. The chemical waste profile sheet provides for identifying any such constituents.

C-2 FREQUENCY OF WASTE ANALYSIS [264.13(a)(3) and (a)(4)]

The solvent mixtures with traces of resins are tested prior to manifesting and shipment using the setaflash test to verify that the mixture does flash under 140°F.

The pH would be run on the waste acids and plating wastes if there is reason to believe that the D002 classification is in error. TCLP analyses may also be performed on plating wastes to determine if TCLP metals are present above regulatory levels.

No additional testing would be done on the filter cake since knowledge of the material being filtered is adequate.

No additional testing would be done on the spent reactor wash solvents since knowledge of the solvents used is adequate to characterize the waste.

Knowledge of the waste material would preclude TCLP testing in most instances. However, TCLP analyses may be performed if it is questionable whether a waste contains constituents above the TCLP regulatory levels.

C-3 INSPECTIONS [264.13(a)(4)]

Drummed waste is inspected before removal from the lab to insure wastes are placed in the proper DOT specified drum (see Process Information - Description of Containers). These procedures along with the chemist's knowledge of the process, will prevent the removal of waste not identified on the internal Ashland Chemical waste profile sheet.

Drums are inspected prior to pumping their contents into the tank to insure that incompatible wastes are not mixed in the tank.

C-4 PARAMETERS AND RATIONALE [122.25(a)(3) and 264.13(b)(1)]

Table 1 contains selected parameters and the rationale for wastes stored at the Ashland facility.

C-5 TEST METHODS [264.13(b)(2), (b)(5), and (b)(6)]

Table 2 contains the parameters and associated test methods.

C-6 SAMPLING METHODS [264.13(b)(3)]

A BelArt Drum Thief of polyethylene material is used to collect samples of drummed waste generated by Ashland. The sampler is 41" long with 3/16 inch openings at each end.

A weighted bottle on a brass chain would be used if the contents of the tank are tested.

TABLE 1

PARAMETER/RATIONALE

	Hazardous Waste	Parameter[1]	Rationale
1.	Solvents Mixture	Flash Point	Majority of solvents used flash at less than 140°F.
2.	Waste Acids	pH	Hazard characteristic is corrosivity which is verified by pH measurement.
3.	Filter cake	Flash Point	Majority of solvents being filtered flash at less than 140°F.
4.	Wash Solvents and Line Flushes	Flash Point Listed Waste	Majority of wash solvents flash at less than 140°F. Components of waste are known.

[1] All wastes are subject to TCLP analyses or generator knowledge of the waste to determine if TCLP constituents are present above regulatory levels.

TABLE 2

Parameter	Test Method	Reference
Flash Point	Setaflash closed cup tester	USEPA SW-846
pH	Electrometric	USEPA SW-846
TCLP constituents	TCLP	40 CFR 268, Appendix I

ASHLAND CHEMICAL INC.
LABORATORY WASTE FROM DUBLIN R&D LABS
WASTE PROFILE SHEET (WPS)

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THIS WASTE PROFILE SHEET IS NEEDED TO ASSIST R&D BUILDING SERVICES IN DISPOSING OF CHEMICAL WASTE AT A MINIMUM COST TO YOU AND TO MEET GOVERNMENTAL REGULATIONS. A WPS IS NEEDED FOR EACH 55 GALLON DRUM.

- Container storage (SO1) is on permitted pad.
- Containers are 55 gallon drums.
- Treatment (mixing with solvents) is in tank 8 (2000 gallon) and/or tank 9 (3000 gallon).
- All treatment is for less than 90 days.

FOR R&D BLDNG.SERV. USE:
WPS:
___Hazardous ___Non-Regulated
EPA Hazardous ID#:
Date to Tanks 8 or 9:
Date Shipped:
Manifest #
Vendor:

YOUR NAME (PRINT):_____PHONE EXT._____

Group: _____Arimax _____IC & S _____Foundry R&D _____Maintenance _____Foundry T.S. _____Pilot Plant _____Polyester _____Research Bldg. Serv. _____Research & Dev. _____SP&A _____VPC/VIC

Date drum filled:_____ Flash Point_____•F

Content: ___Liquid ___Very Viscous ___Solid ___Solid/Liquid Mix

	Chemical Composition	Percent	Trade Name, Exp. Prod.#, Etc.
a.			
b.			
c.			
d.			
e.			
f.			
g.			
	Total	100%	

(Use additional page if necessary)

Does your waste contain any Toxicity Characteristic Constituents listed on the back of this page at or above the regulatory level? _____Yes _____No. If yes, please check back of page.

This is to certify that the chemical composition and information given above is accurate and complete.

_____ _____
Signature(Must be signed by Chemist/Chem. Eng.) Date

Completed forms must be returned to J.W. Boone or M.E. Mullier so that a WPS number can be assigned. You will be given a dated hazardous or non-regulated label which you must apply to the side of the drum. In addition, the WPS number must be spray-painted on the side of the drum. When this is done, the drum must be immediately brought to the Chemical Waste Management area.

October 1990

TCLP CONSTITUENTS AND REGULATORY LEVELS

COMPOUNDS	REG. LEVEL (mg/l)	EPA HW #	PLEASE CHECK
ORGANICS			
Benzene	0.5	D018	____
Carbon tetrachloride	0.5	D019	____
Chlorobenzene	100.0	D021	____
Chloroform	6.0	D022	____
o-Cresol*	200.0	D023	____
m-Cresol*	200.0	D024	____
p-Cresol*	200.0	D025	____
Cresol*	200.0	D026	____
1,4-dichlorobezene	7.5	D027	____
1,2-dichloroethane	0.5	D028	____
1,1-dichloroethylene	0.7	D029	____
2,4-dinitrotoluene	0.13	D030	____
Hexachlorobenzene	0.13	D032	____
Hexachlorol,3-butadiene	0.5	D033	____
Hexachloroethane	3.0	D034	____
Methyl-ethyl-ketone	200.0	D035	____
Nitrobenzene	2.0	D036	____
Pentachlorophenol	100.0	D037	____
Pyridine	5.0	D038	____
Tetrachloroethylene	0.7	D039	____
Trichloroethylene	0.5	D040	____
2,4,5-trichlorophenol	400.0	D041	____
2,4,6-trichlorophenol	2.0	D042	____
Vinyl chloride	0.2	D043	____
METALS			
Arsenic	5.0	D004	____
Barium	100.0	D005	____
Cadmium	1.0	D006	____
Chromium	5.0	D007	____
Lead	5.0	D008	____
Mercury	0.2	D009	____
Selenium	1.0	D010	____
Silver	5.0	D011	____
PESTICIDES			
Chlordane	0.03	D020	____
Endrin	0.02	D012	____
Heptachlor(+ hydroxide)	0.008	D031	____
Lindane	0.4	D013	____
Methoxychlor	10.0	D014	____
Toxaphene	0.5	D015	____
2,4-D	10.0	D016	____
2,4,5-TP (Silvex)	1.0	D017	____

* If o-,m-, & p-cresol cannot be differentiated, the total cresol concentration is used.

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Ashland Chemicals
R&D Labs
Chemical Waste Profile Sheet

Identification Mark on Drum: PE 187

Date Drum Was Filled: 9-28-84 / 10-31-84

Net Weight of Drum Contents: 505 #

DRUM CONTENTS:

Quantity	Trade Name And	Chemical Composition
260#	50405/50400/50423	Polyesters & POLYESTER URETHANES STYRENE
48#	59036/++	MgO w pigments & polyvinyl Acetates
45#	SnFl/C.W.	$CaCO_3$
48#	59019/LP4DA	Adipic POLYESTERS - POLYVINYL ACETATES
104#	50271/50372/Q6585	POLYESTER BASE RESINS

Troy A Wack 10-31-84
Signature and Date

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Ashland Chemicals
R&D Labs
Chemical Waste Profile Sheet

Identification Mark on Drum: FC-206

Date Drum Was Filled: 10-5-84

Net Weight of Drum Contents: 46816.

DRUM CONTENTS:

Quantity	Trade Name	And	Chemical Composition
30 gal	Isolure		Phenolic & solvent
10 gal	Primary Base		Phenolic
10 gal	Furan		Furfural Alcohol
	Small amounts		amine catalyst

Richard L. Pelfrey
Signature and Date

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Ashland Chemicals
R&D Labs
Chemical Waste Profile Sheet

Identification Mark on Drum: FC 206

Date Drum Was Filled: 10/10/84

Net Weight of Drum Contents: 375 lbs

DRUM CONTENTS:

Quantity	Trade Name And Chemical Composition
42 gal	Acrylic Resins in Organic Solvents

H. Randall Shriver 10/10/84
Signature and Date

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Ashland Chemicals
R&D Labs
Chemical Waste Profile Sheet

Identification Mark on Drum: CS-182

Date Drum Was Filled: 10-18-84

Net Weight of Drum Contents:

DRUM CONTENTS:

Quantity	Trade Name	And	Chemical Composition
19 gal			ACETONE
14 gal			MEK
6 gal			MIBK
8 gal			CELLOSOLVE ACETATE
18 gal			ISO PROPYL ACETATE
5 gal			MAK
~ 1 lb			CARBON BLACK (black PIGMENT)
~ ½ lb			TITANIUM DIOXIDE (WHITE PIGMENT)
~2 gal			POLYESTER AND ACRYLIC POLYMER

Maria Rios 10-18-84
Signature and Date

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Ashland Chemicals
R&D Labs
Chemical Waste Profile Sheet

Identification Mark on Drum: CS-227

Date Drum Was Filled: 12/17/84

Net Weight of Drum Contents:

DRUM CONTENTS:

Quantity	Trade Name	And	Chemical Composition
55gal	PSA	reacted monomers - vinyl acetate, ethyl acrylate, 2EHA, acrylic acid, butyl acrylate methyl methacrylate solvents - ethyl acetate, toluene IPA hexane heptane methanol acetone	

Mary A Saint 12/17/84
Signature and Date

FROM USEPA SW 846

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METHOD 1020[1]

SETAFLASH CLOSED-CUP METHOD

1.0 Scope and Application

1.1 Method 1020 make use of the Setaflash® Closed Tester to determine the flash point of paints, enamels, lacquers, varnishes, and related products and their components that have flash points between 0° and 110° C (32° and 230° F) and a viscosity lower than 150 stokes at 25° C (77° F). Tests at higher or lower temperatures are possible.

1.2 The procedures may be used to determine whether a material will or will not flash at a specified temperature or to determine the finite temperature at which a material will flash.

2.0 Summary of Method

2.1 By means of a syringe, 2 ml of sample is introduced through a leakproof entry port into the tightly closed Setaflash Tester or directly into the cup that has been brought to within 3° C (5° F) below the expected flash point.

2.2 As a flash/no flash test, the expected flash point temperature may be a specification (e.g., 60° C). For specification testing, the temperature of the apparatus is raised to the precise temperature of the expected flash point by slight adjustment of the temperature dial. After 1 min, a test flame is applied inside the cup and note is taken as to whether the test sample flashes or not. If a repeat test is necessary, a fresh sample should be used.

2.3 For a finite flash measurement, the temperature is sequentially increased through the anticipated range, the test flame being applied at 5° C (9° F) intervals until a flash is observed. A repeat determination is then made using a fresh sample, starting the test at the temperature of the last interval before the flash point of the material and making tests at increasing 0.5° C (1° F) intervals.

3.0 Interferences

3.1 Ambient pressure, sample homogeneity, drafts, and operator bias can affect flash point values.

---

[1]This method is based on ASTM Method D327-78.

4.0 Apparatus and Materials

4.1 Setaflash Tester as described in Appendix XI of ASTM Method 3278-78.

4.2 Thermometers conforming to specifications given in ASTM Method 3278-78. Test to determine that the scale error does not exceed 0.25° C (0.5° F). A magnifying lens significantly assists in making temperature observations.

4.3 Glass syringe: 2 ± 0.1-ml capacity at 25° C (77° F), to provide a means of taking a uniform sample. Check the capacity by discharging water into a weighing bottle and weighing. Adjust plunger if necessary. A disposable syringe of equal precision may be used.

4.4 Cooling block: Aluminum (described in Appendix X2 of ASTM D3278-78) which fits snugly within the test cup for rapid cooling of the sample cup.

4.5 Barometer.

5.0 Reagents

5.1 p-Xylene: Reference standard for checking the Setaflash Tester.

5.2 Cooling mixture of ice water or dry ice (solid CO_2) and acetone.

5.3 Liquefied petroleum gas.

5.4 Heat transfer paste.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must be collected employing a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 The sample size for each test is 2 ml. Obtain at least a 25-ml sample from the bulk source and store in a nearly full, tightly closed clean glass container or in another container suitable for the type of liquid being sampled.

6.3 Erroneously high flash points may be obtained if precautions are not taken to avoid loss of volatile materials. Do not open sample containers unnecessarily and do not transfer the sample to the cup unless its temperature is at least 10° C (20° F) below the expected flash point. Discard samples in leaky containers.

6.4 Do not use plastic bottles since certain volatile compounds can diffuse through the walls of the bottle.

7.0 Procedure

7.1 Prior to initial use and after removal of the thermometer, insert the thermometer into its pocket with a good heat transfer paste.

7.2 To help in making the necessary settings during a test, determine the relationship between the temperature control dial and thermometer readings at intervals not over 5° C (10° F) throughout the scale range of the heater before the initial use.

7.3 Place the tester in a subdued light and in a position where it is not exposed to disturbing drafts. Provide a black-coated shield, if necessary.

7.4 Read the manufacturer's operating and maintenance instructions on the care and servicing of the tester. Observe the specific suggestions regarding the operation of its various controls.

7.5 Check the accuracy of the tester by determining the flash point of the p-xylene reference standard in duplicate (Appendix X3). The average of the results should be 27.2° ± 0.8° C (81° ± 1.5° F). If not, remove the thermometer and observe whether sufficient heat transfer paste surrounds the thermometer to provide good heat transfer from the cup to the thermometer.

7.6 Ambient to 110° C (230° F).

7.6.1 Inspect the inside of the test cup, lid, and shutter mechanism for cleanliness and freedom from contamination. Use an absorbent tissue to wipe clean, if necessary. Lock the cover lid tightly in place.

7.6.2 Switch the tester on, if not already at stand-by. To rapidly approach the specification flash temperature of the charged sample, turn the heater dial fully clockwise causing the heater signal (red) light to glow. When the thermometer indicates a temperature of about 3° C (5° F) below the specification or target flash point temperature, reduce the heat input to the test cup by slowly turning the heater control dial counter-clockwise until the signal light goes out.
NOTE: When the correct temperature is dialed on the temperature controller, the elapsed time to reach it may be greater than when turned full on, but less attention will be required in the intervening period.
NOTE: The test cup temperature is stable when the signal light slowly cycles on and off.

7.6.3 Determine the barometric pressure to determine the corrected specification temperature at that barometric pressure.

7.6.4 After the test cup temperature has stabilized at the specification or target flash point, charge the syringe with the sample to be tested and transfer the syringe to the filling orifice, taking care not to lose any sample. Discharge the sample into the test cup by depressing

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the syringe plunger to its lowest position, then remove the syringe. If the sample has a viscosity greater than 45 SUS at 37.8° C (100° F) or equivalent of 9.5 cSt at 25° C (77° F), discharge the contents of the syringe directly into the cup. Immediately close tightly the lid and shutter assembly.

7.6.5 Set the 1-min timing device by rotating its knob clockwise to the required setting. In the meantime, open the gas control valve and light the pilot and the test flames. Adjust the test flame size with the pinch valve so as to match the size of the 5/32-in. (4-mm) diameter flame gauge.

7.6.6 After 1 min has elapsed, observe the temperature. If at the specification temperature (accounting for the differences of the barometer reading from 760 mm), apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of approximately 2-1/2 sec. Watch for a flash. (NOTE: The sample is considered to have flashed only if a comparatively large blue flame appears and propagates itself over the surface of the liquid. Occasionally, particularly near the actual flash point temperature, application of the test flame may give rise to a halo; this should be ignored.)

7.6.7 Turn off the test and the pilot flame. Clean the apparatus in preparation for the next test.

7.7 0° C (32° F) to ambient

7.7.1 If the specification or target flash point is at or below ambient temperature, cool the sample to 5° to 10° C (10° to 20° F) below that point by some convenient means.

7.7.2 Cool the tester to the approximate temperature of the sample by inserting the cooling block filled with a cooling mixture into the sample well. Dry the cup with a paper tissue to remove any collected moisture prior to adding the sample. (CAUTION: Be careful in handling the cooling mixture and cooling block; wear gloves and goggles. Mixtures such as dry ice and acetone can produce severe frost bite.) (CAUTION: Be careful in inserting the cooling block into the tester cup to prevent damage to the cup.)

7.7.3 Introduce the sample as in 7.6.4. Allow the temperature to rise under ambient conditions or increase the temperature of the cup by rotating the heater controller clockwise slowly until the specification temperature adjusted for barometric pressure is reached. Determine whether the sample flashes as in 7.6.5 and 7.6.6.

7.7.4 Turn off the test and pilot flames. Clean up the apparatus.

7.8 Ambient to 110° C (230° F)

7.8.1 Preliminary or trial test: Follow steps 7.6.2 to 7.6.5 omitting the barometric reading and using an estimated finite flash point instead of a specification flash point temperature.

7.8.2 After 1 min has elapsed, observe the temperature, apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of 2-1/2 sec. Watch for a flash. (NOTE: The sample is considered to have flashed only if a comparatively large blue flame appears and propagates itself over the surface of the liquid. Occasionally, particularly near the actual flash point temperature, application of the test flame may give rise to a halo; this should be ignored.)

7.8.3 Finite flash point: If a flash is observed, proceed as below.

7.8.3.1 Using a temperature of 5° C (9° F) lower than the temperature observed in 7.8.2, repeat 7.8.1 and 7.8.2. (CAUTION: Be careful in inserting the cooling block into the tester cup to prevent damage to the cup.) If a flash is still observed, repeat at 5° C (9° F) lower intervals until no flash is observed. (NOTE: Never make a repeat test on the same sample. Always take a fresh portion for each test.)

7.8.3.2 Repeat 7.8.1 and 7.8.2 with a new sample, stabilizing the test cup temperature at the temperature at which no flash occurred previously. Observe whether a flash occurs at this temperature. If no flash occurs, increase the temperature at 0.5° C (1° F) intervals by making small incremental adjustment to the temperature controller and allowing 1-min intervals between each increment and the flash point test. Record the temperature at which the flash actually occurs. Record the barometric pressure. Turn off pilot and test flames and clean up tester.

7.8.4 Finite flash point: If no flash point is observed in 7.8.2, proceed as follows.

7.8.4.1 Using a test temperature of 5° C (9° F) higher than the temperature observed in 7.8.2, repeat steps 7.8.1 and 7.8.2. (NOTE: Never make a repeat test on the same sample. Always take a fresh portion for each test.) If no flash is observed, repeat at 5° C (9° F) higher intervals until a flash is observed.

7.8.4.2 Repeat step 7.8.3.2 with a new sample.

7.9 0° C (32° F) to ambient temperature

7.9.1 Preliminary or trial test: Cool the sample to 3° to 5° C (5° to 10° F) below the expected flash point.

7.9.2 Cool the tester to approximately the temperature of the sample by inserting the cooling block filled with a cooling medium into the sample well.

7.9.3 Insert the sample as in 7.6.4. Set the 1-min timing device. After 1 min, apply the test flame by slowly and uniformly opening the side fully and closing completely over a period of approximately 2-1/2 sec. Observe for a flash. Record the temperature.

7.9.4 Finite flash point: If a flash is observed, proceed as follows.

7.9.4.1 Cool a new sample and the sample cup to 5° C (9° F) below the previous temperature (7.9.3). After 1 min, check for a flash as in 7.9.3. If the sample flashes, repeat test at 5° C (9° F) lower intervals until no flash is observed.

7.9.4.2 Repeat with a new sample, cooling both sample and tester to the temperature at which the sample did not flash. After 1 min, observe whether a flash occurs at this temperature. If not, increase the temperature at 0.5° C (1° F) intervals by making small incremental adjustments to the temperature controller, allowing 1 min between each increment and the test for the flash point. Record the temperature at which the flash actually occurs. Record the barometric pressure.

7.9.5 Finite flash point: If no flash point is observed proceed as follows.

7.9.5.1 Using a test temperature of 5° C (9° F) higher than the temperature observed in 7.9.3, repeat step 7.9.3. (CAUTION: Be careful in inserting the cooling block into the tester cup to prevent damage to the cup.) If no flash is observed, repeat at 5° C (9° F) higher intervals until flash is observed.

7.9.5.2 Using a new sample, repeat 7.9.4.2 until a flash occurs. Record the temperature at which the flash occurs and the barometric pressure.

7.10 Cleanup of apparatus and preparation for next test

7.10.1 To prepare for the next test, unlock the lid assembly of the tester and raise to the hinge stop. Soak up liquid samples with an

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absorbent paper tissue and wipe dry. Clean the underside of the lid and filling orifice. A pipe cleaner may be of assistance in cleaning the orifice.

7.10.2 If the sample is a viscous liquid or contains dispersed solids, after soaking up most of the sample add a small amount of a suitable solvent for the sample to the cup and then soak up the solvent and wipe clean the interior surfaces of the cup with an absorbent tissue paper. (NOTE: If necessary to remove residual high boiling solvent residues, moisten tissue with acetone and wipe clean.) (NOTE: If any further cleaning is necessary, remove the lid and shutter assembly. Disconnect the silicone rubber hose and slide the lid assembly to the right to remove. If warm, handle carefully.)

7.10.3 After the cup has been cleaned, its temperature may be rapidly increased to some stand-by value by turning the temperature control dial to an appropriate point. (NOTE: It is convenient to hold the test cup at some stand-by temperature (depending on planned usage) to conserve time in bringing the cup within the test temperature range. The cup temperature may be quickly lowered by inserting the aluminum cooling block filled with an appropriate cooling mixture into the cup.)

7.10.4 The syringe is easily cleaned by filling it several times with acetone or any compatible solvent, discharging the solvent each time, and allowing the syringe to air dry with the plunger removed. Replace the plunger, and pump several times to replace any solvent vapor with air.

7.11 Correction for barometric pressure

7.11.1 When the barometric pressure differs from 760 mm Hg (101.3 kPa), calculate the flash point temperature by means of the following equations:

$$\text{Calculated flash point} = C + 0.03\,(760 - P)$$
$$= F + 0.06\,(760 - P)$$

where:

C, F = observed flash point (°C or °F)

P = barometric pressure (mm Hg).

7.11.2 Likewise determine the corrected specification flash point by the following equation:

$$C = S - 0.03\,(760 - P)$$
$$F = S - 0.06\,(760 - P)$$

where:

C, F = flash point to be observed to obtain the specification flash point at standard pressure (S)

S = specification flash point.

7.12 Report

7.12.1 When using the flash/no flash method, report whether the sample flashed at the required flash point and that the flash/no flash method was used.

7.12.2 If an actual flash point was determined, report the average of duplicate runs to nearest 0.5° C (1° F) provided the difference between the two values does not exceed 1° C (2° F).

8.0 Quality Control

8.1 All quality control data should be available for review.

8.2 Duplicates and standard reference materials should be routinely analyzed.

8.3 The flash point of the p-xylene reference standard must be determined in duplicate at least once per sample batch. The average of the two analyses should be 27° ± 0.8° C (81° ± 1.5° F).

FROM US.EPA SW 846

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METHOD 9040

pH MEASUREMENT

1.0 Scope and Application

1.1 Method 9040 is used to measure the pH of aqueous wastes and those multiphasic wastes where the aqueous phase comprises at least 20% of the total volume of the waste.

2.0 Summary

2.1 The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode. The measuring device is calibrated using a series of solutions of known pH.

3.0 Interferences

3.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

3.2 Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.

3.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.

3.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. It should, therefore, be noted by reporting both the pH and temperature at the time of analysis.

4.0 Apparatus and Materials

4.1 pH Meter: Laboratory or field model. A wide variety of instruments are commercially available with various specifications and optional equipment.

4.2 Glass electrode.

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4.3 Reference electrode: A silver-silver chloride or other reference electrode of constant potential may be used. NOTE: Combination electrodes incorporating both measuring and referenced functions are convenient to use and are available with solid, gel-type filling materials that require minimal maintenance.

4.4 Magnetic stirrer and Teflon-coated stirring bar.

4.5 Thermometer or temperature sensor for automatic compensation.

5.0 Reagents

5.1 Primary standard buffer salts are available from the National Bureau of Standards and should be used in situations where extreme accuracy is necessary. Preparation of reference solutions from these salts requires some special precautions and handling[1] such as low-conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.

5.2 Secondary standard buffers may be prepared from NBS salts or purchased as a solution from commercial vendors. These commercially available solutions have been validated by comparison to NBS standards and are recommended for routine use.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 Samples should be analyzed as soon as possible.

7.0 Procedure

7.1 Calibration

7.1.1 Because of the wide variety of pH meters and accessories, detailed operation procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

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[1]National Bureau of Standards Special Publication 260.

7.1.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Various instrument designs may involve use of a "balance" or "standardize" dial or slope adjustment as outlined in the manufacturer's instructions. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value.

7.2 Place the sample or buffer solution in a clean glass beaker using a sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar. If field measurements are being made, the electrodes may be immersed directly in the sample stream to an adequate depth and moved in a manner to ensure sufficient sample movement across the electrode-sensing element as indicated by drift-free (less than 0.1 pH) readings.

7.3 If the sample temperature differs by more than 2°C from the buffer solution, the measured pH values must be corrected. Instruments are equipped with automatic or manual compensators that electronically adjust for temperature differences. Refer to manufacturer's instructions.

7.4 Thoroughly rinse and gently wipe the electrodes prior to measuring pH of samples. Immerse the electrodes into the sample beaker or sample stream and gently stir at a constant rate to provide homogeneity and suspension of solids. Note and record sample pH and temperature. Repeat measurement on successive volumes of sample until values differ by less than 0.1 pH units. Two or three volume changes are usually sufficient.

8.0 Quality Control

8.1 Duplicate samples and check standards should be analyzed routinely.

8.2 Electrodes must be thoroughly rinsed between samples.

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METHOD 7060

ARSENIC (ATOMIC ABSORPTION, FURNACE TECHNIQUE)

1.0 Scope and Application

1.1 Method 7060 is an atomic absorption procedure approved for determining the concentration of arsenic in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7060, samples must be prepared in order to convert organic forms of arsenic to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to the acid digestion procedure described in this method. Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot of the digestate is spiked with a nickel nitrate solution and is placed manually or by means of an automatic sampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. The absorption of hollow cathode radiation during atomization will be proportional to the arsenic concentration.

2.3 The typical detection limit for this method is 1 µg/l.

3.0 Interferences

3.1 Elemental arsenic and many of its compounds are volatile and therefore samples may be subject to losses of arsenic during sample preparation. Spike samples and relevant standard reference materials should be processed to determine if the chosen dissolution method is appropriate.

3.2 Likewise, caution must be employed during the selection of temperature and times for the dry and char (ash) cycles. A nickel nitrate solution must be added to all digestate prior to analysis to minimize volatilization losses during drying and ashing.

3.3 In addition to the normal interferences experienced during graphite furnace analysis, arsenic analysis can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Arsenic analysis is particularly susceptible to these problems because of its low analytical wavelength (193.7 nm). Simultaneous background correction must be employed to avoid erroneously high results.

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3.4 If the analyte is not completely volatilized and removed from the furnace during atomization, memory effects will occur. If this situation is detected by means of blank burns, the tube should be cleaned by operating the furnace at full power at regular intervals in the analytical scheme.

4.0 Apparatus and Materials

4.1 250-ml Griffin beaker.

4.2 10-ml volumetric flasks.

4.3 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for simultaneous background correction and interfacing with a strip chart recorder.

4.4 Arsenic hollow cathode lamp or electrodeless discharge lamp.

4.5 Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

4.6 Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can easily be recognized.

4.7 Pipets: Microliter with disposable tips. Sizes can range from 5 to 1000 µl as required.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Hydrogen peroxide (30%): Oxidant should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.4 Arsenic standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve

1.320 g of arsenic trioxide (As_2O_3, analytical reagent grade) or equivalent in 100 ml of Type II water containing 4 g NaOH. Acidify the solution with 20 ml conc. HNO_3 and dilute to 1 liter.

5.5 Nickel nitrate solution (5%): Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \cdot 6H_2O$ or equivalent in Type II water and dilute to 100 ml.

5.6 Nickel nitrate solution (1%): Dilute 20 ml of the 5% nickel nitrate to 100 ml with Type II water.

5.7 Arsenic working standards: Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Withdraw appropriate aliquots of the stock solution, add 1 ml of conc. HNO_3, 2 ml of 30% H_2O_2, and 2 ml of the 5% nickel nitrate solution. Dilute to 100 ml with Type II water.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and distilled deionized water. Plastic and glass containers are both suitable.

6.3 Special containers (e.g., containers used for volatile organic analysis) may have to be used if very volatile arsenic compounds are to be analyzed.

6.4 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.5 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared in the manner described in Sections 7.1.1-7.1.3. Sludge-type samples should be prepared according to Method 3050, and samples containing oils, greases, or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample-preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.1.1 Transfer 100 ml of well-mixed sample to a 250-ml Griffin beaker, add 2 ml of 30% H_2O_2 and sufficient conc. HNO_3 to result in an acid concentration of 1% (v/v). Heat for 1 hr at 95° C or until the volume is slightly less than 50 ml.

7.1.2 Cool and bring back to 50 ml with Type II water.

7.1.3 Pipet 5 ml of this digested solution into a 10-ml volumetric flask, add 1 ml of the 1% nickel nitrate solution and dilute to 10 ml with Type II water. The sample is now ready for injection into the furnace.

7.2 The 193.7-nm wavelength line and a background correction system must be employed. Follow the manufacturer's suggestions for all other spectrophotometer parameters.

7.3 Furnace parameters suggested by the manufacturer should be employed as guidelines. Since temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

7.4 Inject a measured µl aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

7.5 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.6 Run a check standard after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced.

7.7 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

7.8 Duplicates, spiked samples, and check standards should be routinely analyzed.

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7061

ARSENIC (ATOMIC ABSORPTION, GASEOUS HYDRIDE)

1.0 Scope and Application

1.1 Method 7061 is an atomic absorption procedure for determining the concentration of arsenic in wastes, mobility procedure extracts, soils, and groundwater. Method 7061 is approved only for sample matrices that do not contain high concentrations of chromium, copper, mercury, nickel, silver, cobalt, and molybdenum. All samples must be subjected to an appropriate solution step prior to analysis. Spiked samples and relevant standard reference reference materials are employed to determine the applicability of the method to a given waste.

2.0 Summary of Method

2.1 Samples are prepared according to the nitric/sulfuric acid digestion procedure described in this method. Next, the arsenic in the digestate is reduced to the trivalent form using tin chloride. The trivalent arsenic is then converted to a volatile hydride using hydrogen produced from a zinc/HCl reaction.

2.2 The volatile hydride is swept into an argon-hydrogen flame located in the optical path of an atomic absorption spectrophotometer. The resulting absorption of the hollow cathode radiation is proportional to the arsenic concentration.

2.3 The typical detection limit for this method is 0.002 mg/l.

3.0 Interferences

3.1 High concentrations of chromium, cobalt, copper, mercury, molybdenum, nickel, and silver can cause analytical interferences.

3.2 Traces of nitric acid left following the sample workup can result in analytical interferences. Nitric acid must be distilled off by heating the sample until fumes of SO_3 are observed.

3.3 Elemental arsenic and many of its compounds are volatile and therefore certain samples may be subject to losses of arsenic during sample preparation.

4.0 Apparatus and Materials

4.1 100-ml beaker.

4.2 Electric hot plate.

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4.3 A commercially available zinc slurry/hydride generator or a generator constructed from the following materials (see Figure 1).

4.3.1 Medicine dropper that can be fitted into a size "0" rubber stopper and that is capable of delivering 1.5 ml.

4.3.2 50-ml pear-shaped reaction flask with two 14/20 necks (Scientific Glass JM-5835).

4.3.3 Gas inlet-outlet tube constructed from a micro cold-finger condenser (JM-3325) by cutting the portion below the 14/20 ground glass joint.

4.3.4 Magnetic stirrer to homogenize the zinc slurry.

4.3.5 10-cm polyethylene drying tube filled with glass to prevent particulate matter from entering the burner.

4.3.6 Flow meter capable of measuring 1 liter/minute.

4.4 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for interfacing with a strip chart recorder.

4.5 Burner recommended by the particular instrument manufacturer for the argon-hydrogen flame.

4.6 Arsenic hollow cathode lamp or arsenic electrodeless discharge lamp.

4.7 Strip chart recorder.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Concentrated sulfuric acid: Acid should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.4 Concentrated hydrochloric acid: Acid should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

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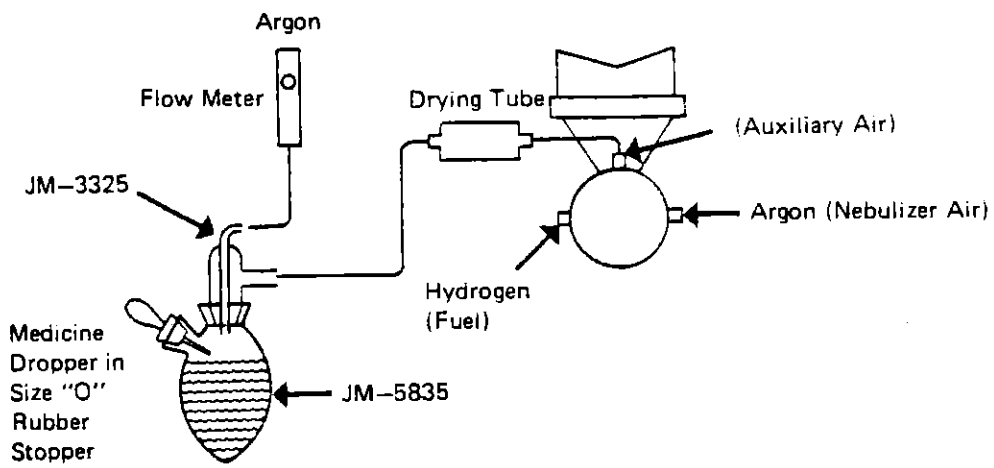

Figure 1. Zinc slurry hydride generator apparatus set-up and AAS sample introduction system.

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5.5 Diluent: Add 100 ml 18 N H_2SO_4 and 400 ml concentrated HCl to 400 ml Type II water and dilute to a final volume of 1 liter with Type II water.

5.6 Potassium iodide solution: Dissolve 20 g KI in 100 ml Type II water.

5.7 Stannous chloride solution: Dissolve 100 g $SnCl_2$ in 100 ml conc. HCl.

5.8 Arsenic solutions

5.8.1 Arsenic standard solution (1,000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products or Fisher Scientific) and verify by comparison with a second standard, or dissolve 1.320 g of arsenic trioxide As_2O_3 (analytical reagent grade) or equivalent in 100 ml of Type II water containing 4 g NaOH. Acidify the solution with 20 ml conc. HNO_3 and dilute to 1 liter.

5.8.2 Intermediate arsenic solution: Pipet 1 ml stock arsenic solution into a 100-ml volumetric flask and bring to volume with deionized distilled water containing 1.5 ml concentrated HNO_3/liter (1 ml = 10 µg As).

5.8.3 Standard arsenic solution: Pipet 10 ml intermediate arsenic solution into a 100-ml volumetric flask and bring to volume with deionized distilled water containing 1.5 ml concentrated HNO_3/liter (1 ml = 1 µg As).

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and distilled deionized water. Plastic and glass containers are both suitable.

6.3 Special containers (e.g., containers used for volatile organic analysis) may have to be used if very volatile arsenic compounds are to be analyzed.

6.4 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.5 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Place a 50-g sample (or, in the case of analysis of EP extracts, 50 ml) of the material to be analyzed in a 100-ml beaker. Add 10 ml conc.

HNO_3 and 12 ml 18 N H_2SO_4. Evaporate the sample in the hood on an electric hot plate until white SO_3 fumes are observed (a volume of about 20 ml). Do not let the sample char. If charring occurs, immediately turn off the heat, cool, and add an additional 3 ml of HNO_3. Continue to add additional HNO_3 in order to maintain an excess (as evidenced by the formation of brown fumes). Do not let the solution darken, because arsenic may be reduced and lost. When the sample remains colorless or straw yellow during evolution of SO_3 fumes, the digestion is complete. Cool the sample, add about 25 ml Type II water, and again evaporate until SO_3 fumes are produced in order to expel oxides of nitrogen. Cool. Transfer the digested sample to a 100-ml volumetric flask. Add 40 ml of concentrated HCl and bring to volume with Type II water.

7.2 Prepare working standards from the standard arsenic solution. Transfer 0, 0.5, 1.0, 1.5, 2.0, and 2.5 ml of standard to 100-ml volumetric flasks and bring to volume with diluent. These concentrations will be 0, 5, 10, 15, 20, and 25 µg As/liter.

7.3 If EP extracts are being analyzed or if a matrix interference is encountered, take the 15-, 20-, and 25-mg/liter standards and quantitatively transfer 25 ml from each of these standards into separate 50-ml volumetric flasks. Add 10 ml of the prepared sample to each flask. Bring to volume with Type II water containing 1.5 ml HNO_3/liter.

7.4 Add 10 ml of prepared sample to a 50-ml volumetric flask. Bring to volume with Type II water containing 1.5 ml HNO_3/liter. This is the blank.

> NOTE: The absorbance from the blank will be one-fifth that produced by the prepared sample. The absorbance from the spiked standards will be one-half that produced by the standards plus the contribution from one-fifth of the prepared sample. Keeping these in mind, the correct dilutions to produce optimum absorbance can be judged.

7.5 Transfer a 25-ml portion of the digested sample or standard to the reaction vessel, and add 1 ml potassium iodide solution. Add 0.5 ml $SnCl_2$ solution. Allow at least 10 min for the metal to be reduced to its lowest oxidation state. Attach the reaction vessel to the special gas inlet-outlet glassware. Fill the medicine dropper with 1.50 ml zinc slurry that has been kept in suspension with the magnetic stirrer. Firmly insert the stopper containing the medicine dropper into the side neck of the reaction vessel. Squeeze the bulb to introduce the zinc slurry into the sample or standard solution. The metal hydride will produce a peak almost immediately. After the recorder pen begins to return to the base line, the reaction vessel can be removed. CAUTION: Arsine is very toxic. Precautions must be taken to avoid inhaling arsine gas.

7.6 Use the 193.7-nm wavelength and background correction for the analysis of arsenic.

7.7 Follow the manufacturer's instructions for operating an argon hydrogen flame. The argon-hydrogen flame is colorless, so it may be useful to aspirate a low concentration of sodium to ensure that ignition has occurred.

7.8 If the method of standard additions was employed, plot the absorbances of spiked samples and blank vs. the concentrations. The extrapolated value will be one-tenth the concentration of the original sample. If the plot does not result in a straight line, a nonlinear interference is present. This problem can sometimes be overcome by dilution, or addition of other reagents if there is some knowledge about the waste. If the method of standard additions was not required then the concentration can be part of the calibration curve.

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7080

BARIUM (ATOMIC ABSORPTION, DIRECT ASPIRATION METHOD)

1.0 Scope and Application

1.1 Method 7080 is an atomic absorption procedure approved for determining the concentration of barium in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7080, samples must be prepared for direct aspiration. The method of sample preparation will vary according to the sample matrix. Aqueous samples are subjected to an acid digestion procedure (Method 3010). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, an ionization suppressant is added and a representative aliquot is aspirated into a nitrous oxide/acetylene flame. The resulting absorption of hollow cathode radiation will be proportional to the barium concentration. Background correction must be employed.

2.3 The typical detection limit for this method is 0.1 mg/l; typical sensitivity is 0.4 mg/l.

3.0 Interferences

3.1 High hollow cathode current settings and a narrow spectral band pass must be used since both barium and calcium emit strongly at barium's analytical wavelength.

3.2 Barium undergoes significant ionization in the nitrous oxide/acetylene flame, resulting in a significant decrease in sensitivity. Therefore an ionization suppressant must be added to both standards and samples.

3.3 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

3.4 If an air/acetylene flame is used, then the presence of phosphate silicon and aluminum will decrease the sensitivity. This problem can be overcome by adding a releasing agent (e.g., lanthanum) to both samples and standard.

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4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: single or dual channel, single- or double-beam instrument, having a grating monochromator, photomultiplier detector, adjustable slits, and provisions for background correction.

4.2 Barium hollow cathode lamp or electrodeless discharge lamp.

4.3 Strip chart recorder (optional).

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Barium standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 1.787 g barium chloride ($BaCl_2 \cdot 2H_2O$ (analytical reagent grade) in Type II water and dilute to 1 liter.

5.4 Potassium chloride solution: Dissolve 95 g potassium chloride (KCl) in Type II water and dilute to 1 liter.

5.5 Lanthanum chloride solution if needed: Dissolve 25 g reagent grade La_2O_3 slowly in 250 ml concentracted HCl. (Reaction can be violent.) Dilute to 500 ml with Type II water.

5.6 Barium working standards: Prepare dilutions of the stock barium solution to be used as calibration standards. To each 100 ml of standard and sample add 2.0 ml potassium chloride solution.

5.7 Air: Cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or a cylinder of industrial-grade compressed air.

5.8 Acetylene: Should be of high purity. Acetone, which is usually present in acetylene cylinders, can be prevented from entering and affecting flame conditions by replacing the cylinder before the pressure has fallen to 50 psig.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3010; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 553.6-nm wavelength line shall be used.

7.3 A fuel-rich nitrous oxide/acetylene flame shall be used.

7.4 Follow the manufacturer's operating instructions for all other instrument parameters.

7.5 Either (1) run a series of barium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.6 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.7 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.8 The final calculated concentration should take into account all dilution and concentration factors.

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8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7081

BARIUM (ATOMIC ABSORPTION, FURNACE METHOD)

1.0 Scope and Application

1.1 Method 7081 is an atomic absorption procedure approved for determining the concentration of barium in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7081, samples must be prepared in order to convert organic forms of barium to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to an acid digestion procedure (Method 3020). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is placed manually or by means of an automatic sampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. The absorption of hollow cathode radiation during atomization will be proportional to the barium concentration.

2.3 The typical detection limit for this method is 2 µg/l.

3.0 Inferences

3.1 Barium is known to form a barium carbide in the graphite furnace. This less volatile carbide can cause losses of sensitivity and memory effects.

3.2 The long residence time and the high concentration of the analyte in the optical path of the graphite furnace can lead to severe physical and chemical interferences. Furnace parameters must be optimized to minimize these effects.

3.3 Because of possible chemical interaction, nitrogen should not be used as a purge gas.

3.4 Halide acids should not be used.

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4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for interfacing with a strip chart recorder.

4.2 Barium hollow cathode lamp or electrodeless discharge lamp.

4.3 Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

4.4 Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can easily be recognized.

4.5 Pipets: Microliter with disposable tips. Sizes can range from 5 to 1000 µl as required.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Barium standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 1.787 g barium chloride ($BaCl_2 \cdot 2H_2O$, analytical reagent grade) in Type II water and dilute to 1 liter.

5.4 Potassium chloride solution: Dissolve 95 g potassium chloride (KCl) in Type II water and dilute to 1 liter.

5.5 Barium working standards: Prepare dilution of the stock barium solution to be used as calibration standards. To each 100 ml of standard and sample add 2.0 ml potassium chloride solution.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3020; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 553.6-nm line is the analytical wavelength to be used for barium analysis.

7.3 Follow the manufacturer's operating instructions for all other spectrophotometer parameters.

7.4 Furnace parameters suggested by the manufacturer should be employed as guidelines. Since temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

7.5 Inject a measured µl aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

7.6 Either (1) run a series of barium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.7 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.8 Run a check standard after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced.

7.9 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7130

CADMIUM (ATOMIC ABSORPTION, DIRECT ASPIRATION METHOD)

1.0 Scope and Application

1.1 Method 7130 is an atomic absorption procedure approved for determining the concentration of cadmium in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7130, samples must be prepared for direct aspiration. The method of sample preparation will vary according to the sample matrix. Aqueous samples are subjected to acid digestion procedure (Method 3010). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is aspirated into an air/acetylene flame. The resulting absorption of hollow cathode radiation will be proportional to the cadmium concentration. Background correction must be employed for all analyses.

2.3 The typical detection limit for this method is 0.005 mg/l; typical sensitivity is 0.025 mg/l.

3.0 Interferences

3.1 Nonspecific absorption and light scattering can be significant at the analytical wavelength. Thus background correction is required.

3.2 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument, having a grating monochromator, photomultiplier detector, adjustable slits, and provisions for background correction.

4.2 Cadmium hollow cathode lamp or electrodeless discharge lamp.

4.3 Strip chart recorder (optional).

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5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Cadmium standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 2.282 g cadmium sulfate ($CdSO_4 \cdot 8H_2O$, analytical reagent grade) and dissolve in Type II water or equivalent.

5.4 Cadmium working standards: These standards should be prepared with the same type and same concentration of acid that will be found in the analytical solution.

5.5 Air: Cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or a cylinder of industrial-grade compressed air.

5.6 Acetylene: Should be of high purity. Acetone, which is usually present in acetylene cylinders, can be prevented from entering and affecting flame conditions, by replacing the cylinder before the pressure has fallen to 50 psig.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3010; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according

to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 228.8-nm wavelength line and background correction shall be employed.

7.3 An oxidizing air/acetylene flame shall be used.

7.4 Follow the manufacturer's operating instructions for all other instrument parameters.

7.5 Either (1) run a series of cadmium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.6 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.7 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.8 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

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8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7131

CADMIUM (ATOMIC ABSORPTION, FURNACE METHOD)

1.0 Scope and Application

1.1 Method 7131 is an atomic absorption procedure approved for determining the concentration of cadmium in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7131, samples must be prepared in order to convert organic forms of cadmium to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to an acid digestion procedure (Method 3020). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is placed manually or by means of an automatic sampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. The absorption of hollow cathode radiation during atomization will be proportional to the cadmium concentration.

2.3 The typical detection limit for this method is 0.1 µg/l.

3.0 Interferences

3.1 The long residence time and high concentrations of the atomized sample in the optical path of the graphite furnace can result in severe physical and chemical interferences. Furnace parameters must be optimized to minimize these effects.

3.2 In addition to the normal interferences experienced during graphite furnace analysis, cadmium analysis can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Simultaneous background correction must be employed to avoid erroneously high results.

3.3 If the analyte is not completely volatilized and removed from the furnace during atomization, memory effects will occur. If this situation is detected, the tube should be cleaned by operating the furnace at higher atomization temperatures.

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4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for simultaneous background correction and interfacing with a strip chart recorder.

4.2 Cadmium hollow cathode lamp or electrodeless discharge lamp.

4.3 Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

4.4 Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can easily be recognized.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Ammonium phosphate solution (40%): Dissolve 40 g of ammonium phosphate, $(NH_4)2HPO_4$ (analytical reagent grade), in Type II water and dilute to 100 ml.

5.4 Cadmium standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 2.282 g of cadmium sulfate (3 $CdSO_4 \cdot 8H_2O$), analytical reagent grade), in Type II water and dilute to 1 liter.

5.5 Cadmium working standards: These standards should be prepared with the same type and same concentration of acid that will be found in the analytical solution.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedures

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3020; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 228.8-nm wavelength line and background correction shall be used.

7.3 Follow the manufacturer's operating instructions for all other spectrophotometer parameters.

7.4 Furnace parameters suggested by the manufacturer should be employed as guidelines. Since temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

7.5 Inject a measured µl aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

7.6 For certain sample types the addition of 2 ml of an ammonium phosphate solution to 100 ml of standards and samples will elevate charring (ashing) temperatures which may eliminate matrix interferences. Ammonium sulfate, $(NH_4)_2SO_4$, has been reported to have a similar effect on these samples.

7.7 Either (1) run a series of cadmium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.8 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

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7.9 Run a check standard after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced.

7.10 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.11 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7190

CHROMIUM (ATOMIC ABSORPTION, DIRECT ASPIRATION METHOD)

1.0 Scope and Application

1.1 Method 7190 is an atomic absorption procedure approved for determining the concentration of chromium in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7190, samples must be prepared for direct aspiration. The method of sample preparation will vary according to the sample matrix. Aqueous samples are subjected to an acid digestion procedure (Method 3010). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is aspirated into an nitrous oxide/acetylene flame. The resulting absorption of hollow cathode radiation will be proportional to the chromium concentration.

2.3 The typical detection limit for this method is 0.05 mg/l; typical sensitivity is 0.25 mg/l.

3.0 Interferences

3.1 The nitrous oxide/acetylene flame is the recommended flame since chromium analysis in an air/acetylene flame suffers from matrix interferences caused by nickel iron and other metals. If an air/acetylene flame must be used it should be lean.

3.2 An ionization interference may occur in the nitrous oxide flame if the samples have a significantly higher amount of alkali salts than the standards. If this interference is encountered, an ionization suppressant should be added to both samples and standards.

3.3 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument, having a grating monochromator, photomultiplier detector, adjustable slits, and provisions for background correction.

4.2 Chromium hollow cathode lamp or electrodeless discharge lamp.

4.3 Strip chart recorder (optional).

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Chromium standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 1.923 g of chromium trioxide (CrO_3, reagent grade) in Type II water, acidify with redistilled HNO_3 and dilute to 1 liter.

5.4 Chromium working standards: These standards should be prepared with the same type and same concentration of acid that will be found in the analytical solution.

5.5 Nitrous oxide cylinder.

5.6 Air: Cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or a cylinder of industrial-grade compressed air.

5.7 Acetylene: Should be of high purity. Acetone, which is usually present in acetylene cylinders, can be prevented from entering and affecting flame conditions, by replacing the cylinder before the pressure has fallen to 50 psig.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3010; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 357.9-nm wavelength line and background correction shall be employed.

7.3 A fuel-rich nitrous oxide/acetylene flame shall be used.

7.4 Follow the manufacturer's operating instructions for all other instrument parameters.

7.5 Either (1) run a series of chromium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.6 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.7 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.8 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

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8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7191

CHROMIUM (ATOMIC ABSORPTION, FURNACE METHOD)

1.0 Scope and Application

1.1 Method 7191 is an atomic absorption procedure approved for determining the concentration of chromium in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7191, samples must be prepared in order to convert organic forms of chromium to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to an acid digestion procedure (Method 3020). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is placed manually or by means of an automatic sampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. The absorption of hollow cathode radiation during atomization will be proportional to the chromium concentration.

2.3 The typical detection limit for this method is 1 µg/l.

3.0 Interferences

3.1 The long residence time and high concentrations of the atomized sample in the optical path of the graphite furnace can result in severe physical and chemical interferences. Furnace parameters must be optimized to minimize these effects.

3.2 If the analyte is not completely volatilized and removed from the furnace during atomization, memory effects will occur. If this situation is detected, the tube should be cleaned by operating the furnace at higher atomization temperatures.

3.3 Nitrogen should not be used as the purge gas because of a possible CN band interference.

3.4 Low concentrations of calcium may cause interferences; at concentrations above 200 mg/l, calcium's effect is constant. Calcium nitrate (see Section 5.4) is therefore added to ensure a known constant effect.

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4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for simultaneous background correction and interfacing with a strip chart recorder.

4.2 Chromium hollow cathode lamp or electrodeless discharge lamp.

4.3 Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

4.4 Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can easily be recognized.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Hydrogen peroxide (30%).

5.4 Calcium nitrate solution: Dissolve 11.8 g of calcium nitrate, $Ca(NO_3)_2 \cdot 4H_2O$ (analytical reagent grade), in Type II water and dilute to 1 liter.

5.5 Chromium standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 1.923 g of chromium trioxide (CrO_3, analytical reagent grade) in Type II water and dilute to 1 liter.

5.6 Chromium working standards: These standards should be prepared to contain 0.5% (v/v) HNO_3; 1 ml of 30% H_2O_2 and 1 ml of the calcium nitrate solution may be added to lessen interferences.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedures

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3020; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 357.9-nm wavelength line shall be used.

7.3 Follow the manufacturer's operating instructions for all other spectrophotometer parameters.

7.4 Furnace parameters suggested by the manufacturer should be employed as guidelines. Since temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

7.5 Inject a measured µl aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

7.6 Either (1) run a series of chromium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.7 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

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7.8 Run a check standard after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced.

7.9 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.10 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7420

LEAD (ATOMIC ABSORPTION, DIRECT ASPIRATION METHOD)

1.0 Scope and Application

1.1 Method 7420 is an atomic absorption procedure approved for determining the concentration of lead in wastes, mobility procedure extracts, and soils. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7420, samples must be prepared for direct aspiration. The method of sample preparation will vary according to the sample matrix. Aqueous samples are subjected to an acid digestion procedure (Method 3010). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is aspirated into an air/acetylene flame. The resulting absorption of hollow cathode radiation will be proportional to the lead concentration. Background correction must be employed for all analyses.

2.3 The typical detection limit for this method is 0.1 mg/l; typical sensitivity is 0.5 mg/l.

3.0 Interferences

3.1 Background correction is required since nonspecific absorption and light scattering can be significant at the analytical wavelength.

3.2 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument, having a grating monochromator, photomultiplier detector, adjustable slits, and provisions for background correction.

4.2 Lead hollow cathode lamp or electrodeless discharge lamp.

4.3 Strip chart recorder (optional).

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5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Lead standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 1.599 g of lead nitrate, $Pb(NO_3)_2$ (analytical reagent grade), and dissolve in Type II water, acidify with 10 ml redistilled HNO_3, and dilute to 1 liter with Type II water.

5.4 Lead working standards: These standards should be prepared with the same type and same concentration of acid that will be found in the analytical solution.

5.5 Air: Cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or a cylinder of industrial-grade compressed air.

5.6 Acetylene: Should be of high purity. Acetone, which is usually present in acetylene cylinders, can be prevented from entering and affecting flame conditions, by replacing the cylinder before the pressure has fallen to 50 psig.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3010; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according

to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 283.3-nm wavelength line and background correction shall be used.

7.3 An oxidizing air/acetylene flame shall be used.

7.4 Follow the manufacturer's operating instructions for all other instrument parameters.

7.5 Either (1) run a series of lead standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.6 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.7 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.8 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

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8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7421

LEAD (ATOMIC ABSORPTION, FURNACE METHOD)

1.0 Scope and Application

1.1 Method 7421 is an atomic absorption procedure approved for determining the concentration of lead in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7421, samples must be prepared in order to convert organic forms of lead to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to an acid digestion procedure (Method 3020). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is placed manually or by means of an automatic sampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. The absorption of hollow cathode radiation during atomization will be proportional to the lead concentration.

2.3 The typical detection limit for this method is 1 µg/l.

3.0 Interferences

3.1 The long residence time and high concentrations of the atomized sample in the optical path of the graphite furnace can result in severe physical and chemical interferences. Furnace parameters must be optimized to minimize these effects.

3.2 In addition to the normal interferences experienced during graphite furnace analysis, lead analysis can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Simultaneous background correction must be employed to avoid erroneously high results.

3.3 If the analyte is not completely volatilized and removed from the furnace during atomization, memory effects will occur. If this situation is detected, the tube should be cleaned by operating the furnace at higher atomization temperatures.

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3.4 Sulfate can suppress lead absorbance. This interference can be eliminated or lessened by adding a lanthanum releasing agent (10 ml lanthanum solution per 100 ml of solution).

4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for simultaneous background correction and interfacing with a strip chart recorder.

4.2 Lead hollow cathode lamp or electrodeless discharge lamp.

4.3 Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

4.4 Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can easily be recognized.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Lanthanum nitrate solution: Dissolve 58.64 g of ACS reagent grade La_2O_3 in 100 ml concentrated HNO_3 and dilute to 1000 ml with Type II water.

5.4 Lead standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 1.599 g lead nitrate, $Pb(NO_3)_2$ (analytical reagent grade), in Type II water and acidify with 10 ml redistilled HNO_3. Dilute to 1 liter.

5.5 Lead working standards: These standards should be prepared such that they contain 0.5% (v/v) HNO_3.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedures

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3020; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 283.3-nm wavelength line and background correction shall be used.

7.3 Follow the manufacturer's operating instructions for all other spectrophotometer parameters.

7.4 Furnace parameters suggested by the manufacturer should be employed as guidelines. Since temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

7.5 Inject a measured µl aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be dilated in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

7.6 Either (1) run a series of lead standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.7 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

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7.8 If sulfate interference is encountered, add 10 ml of lanthanum solution to each 100 ml of solution.

7.9 Run a check standard after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced.

7.10 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.11 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7470

MERCURY (MANUAL COLD-VAPOR TECHNIQUE)

1.0 Scope and Application

1.1 Method 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility procedure extracts, aqueous wastes and groundwaters. (Method 7470 can also be used for analyzing certain solid and sludge-type wastes; however, Method 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis, the samples must be prepared according to the procedure discussed in this method.

2.2 Method 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for this method is 0.0002 mg/l.

3.0 Interferences

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/l of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from Type II water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/l had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 ml) since, during the oxidation step, chlorides are converted to free chlorine which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 ml). In addition, the dead air space in the BOD bottle must be purged before adding stannous sulfate. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

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3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer or equivalent: Any atomic absoption unit having an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed. Instruments designed specifically for the measurement of mercury using the cold-vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

4.2 Mercury hollow cathode lamp or electrodeless discharge lamp.

4.3 Recorder: Any multirange variable speed recorder that is compatible with the UV detection system is suitable.

4.4 Absorption cell: Standard spectrophotometer cells 10 cm long having quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 1 in. O.D. x 4.5 in. The ends are ground perpendicular to the longitudinal axis and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2-in. x 2-in. cards. One-in.-diameter holes are cut in the middle of each card. The cards are then placed over each end of the cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

4.5 Air pump: Any peristaltic pump capable of delivering 1 liter air/min may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

4.6 Flowmeter: Capable of measuring an air flow of 1 liter/min.

4.7 Aeration tubing: A straight glass frit having a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.

4.8 Drying tube: 6-in. x 3/4-in.-diameter tube containing 20 g of magnesium perchlorate or a small reading lamp with 60-W bulb which may be used to prevent condensation of moisture inside the cell. The lamp should be positioned to shine on the absorption cell so that the air temperature in the cell is about 10° C above ambient.

4.9 The cold-vapor generator is assembled as shown in Figure 1.

4.10 The apparatus shown in Figure 1 is a closed system. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system.

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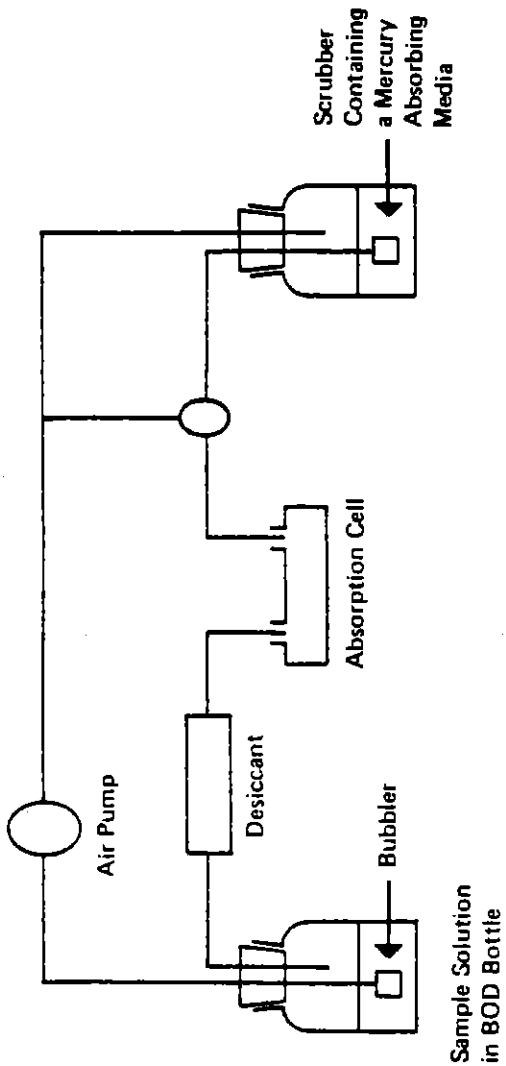

Figure 1. Apparatus for flameless mercury determination.

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4.11 Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system to either vent the mercury vapor into an exhaust hood or pass the vapor through some absorbing media, such as:

1. equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4

2. 0.25% iodine in a 3% KI solution

A specially treated charcoal that will adsorb mercury vapor is also available from Barnebey and Cheney, E. 8th Ave. and N. Cassidy St., Columbus, Ohio 43219, Cat. #580-13 or #580-22.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Sulfuric acid, conc.: Reagent grade.

5.3 Sulfuric acid, 0.5 N: Dilute 14.0 ml of conc. sulfuric acid to 1.0 liter.

5.4 Nitric acid, conc.: Reagent grade of low mercury content. If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

5.5 Stannous sulfate: Add 25 g stannous sulfate to 250 ml of 0.5 N sulfuric acid. This mixture is a suspension and should be stirred continuously during use. (Stannous chloride may be used in place of stannous sulfate.)

5.6 Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in Type II water and dilute to 100 ml. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)

5.7 Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 ml of Type II water.

5.8 Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate in 100 ml of Type II water.

5.9 Stock mercury solution: Dissolve 0.1354 g of mercuric chloride in 75 ml of Type II water. Add 10 ml of conc. HNO_3 and adjust the volume to 100.0 ml (2 ml = 1 mg Hg).

5.10 Mercury working standard: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 µg per ml. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask as needed before addition of the aliquot.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid. The suggested maximum holding times for these samples are 38 days in glass containers and 13 in plastic containers.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Transfer 100 ml, or an aliquot diluted to 100 ml, containing not more than 1.0 µg of mercury, to a 300-ml BOD bottle. Add 5 ml of sulfuric acid and 2.5 ml of conc. nitric acid, mixing after each addition. Add 15 ml of potassium permanganate solution to each sample bottle. Sewage samples may require additional permanganate. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 min. Add 8 ml of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95° C. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. After a delay of at least 30 sec, add 5 ml of stannous sulfate and immediately attach the bottle to the aeration apparatus and continue as described in Section 7.3.

7.2 Standard preparation: Transfer 0-, 0.5-, 1.0-, 2.0-, 5.0-, and 10.0-ml aliquots of the mercury working standard containing 0 to 1.0 µg of mercury to a series of 300-ml BOD bottles. Add enough Type II water to each bottle to make a total volume of 100 ml. Mix thoroughly and add 5 ml of conc. sulfuric acid and 2.5 ml of conc. nitric acid to each bottle. Add 15 ml of $KMnO_4$ solution to each bottle and allow to stand at least 15 min. Add 8 ml of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95° C. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. When the solution has been decolorized, wait 30 sec, add 5 ml of the stannous sulfate solution, and immediately attach the bottle to the aeration apparatus and continue as described in Section 7.3.

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7.3 Analysis: At this point the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 liter/min, is allowed to run continuously. The absorbance will increase and reach a maximum within 30 sec. As soon as the recorder pen levels off (approximately 1 min), open the bypass valve and continue the aeration until the absorbance returns to its minimum value. Close the bypass valve, remove the stopper and frit from the BOD bottle, and continue the aeration.

7.4 Construct a calibration curve by plotting the absorbance of standards versus micrograms of mercury. Determine the peak height of the unknown from the chart and read the mercury value from the standard curve.

7.5 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.6 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.7 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7471

MERCURY IN SOLID OR SEMISOLID WASTE (MANUAL COLD-VAPOR TECHNIQUE)

1.0 Scope and Application

1.1 Method 7471 is approved for measuring total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis the samples must be prepared according to the procedures discussed in this method.

2.2 Method 7471, a cold-vapor atomic absorption method, is based on the absorption of radiation at the 253.7-nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for this method is 0.0002 mg/l.

3.0 Interferences

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/l of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from Type II water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/l had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 ml) since, during the oxidation step, chlorides are converted to free chlorine which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 ml). In addition, the dead air space in the BOD bottle must be purged before adding stannous sulfate. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer or equivalent: Any atomic absoption unit having an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed. Instruments designed specifically for the measurement of mercury using the cold-vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

4.2 Mercury hollow cathode lamp or electrodeless discharge lamp.

4.3 Recorder: Any multirange variable speed recorder that is compatible with the UV detection system is suitable.

4.4 Absorption cell: Standard spectrophotometer cells 10 cm long having quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 1 in. O.D. x 4.5 in. The ends are ground perpendicular to the longitudinal axis and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2-in. x 2-in. cards. One-in.-diameter holes are cut in the middle of each card. The cards are then placed over each end of the cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

4.5 Air pump: Any peristaltic pump capable of delivering 1 liter air/min may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

4.6 Flowmeter: Capable of measuring an air flow of 1 liter/min.

4.7 Aeration tubing: A straight glass frit having a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.

4.8 Drying tube: 6-in. x 3/4-in.-diameter tube containing 20 g of magnesium perchlorate or a small reading lamp with 60-W bulb which may be used to prevent condensation of moisture inside the cell. The lamp should be positioned to shine on the absorption cell so that the air temperature in the cell is about 10° C above ambient.

4.9 The cold-vapor generator is assembled as shown in Figure 1.

4.10 The apparatus shown in Figure 1 is a closed system. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system.

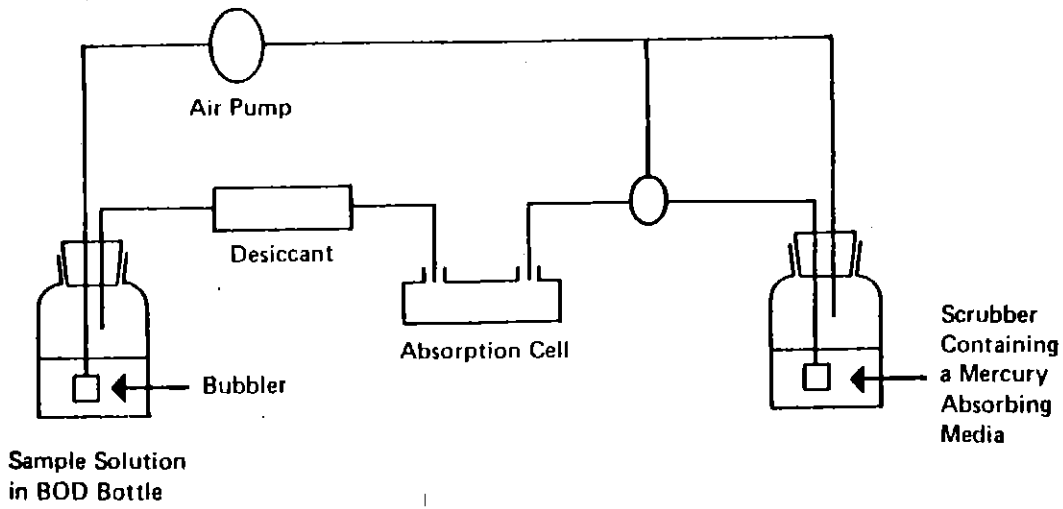

Figure 1. Apparatus for flameless mercury determination.

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4.11 Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system to either vent the mercury vapor into an exhaust hood or pass the vapor through some absorbing media, such as:

1. equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4

2. 0.25% iodine in a 3% KI solution

A specially treated charcoal that will adsorb mercury vapor is also available from Barnebey and Cheney, E. 8th Ave. and N. Cassidy St., Columbus, Ohio 43219, Cat. #580-13 or #580-22.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Aqua regia: Prepare immediately before use by carefully adding three volumes of conc. HCl to one volume of conc. HNO_3.

5.3 Sulfuric acid, 0.5 N: Dilute 14.0 ml of conc. sulfuric acid to 1 liter.

5.4 Stannous sulfate: Add 25 g stannous sulfate to 250 ml of 0.5 N sulfuric acid. This mixture is a suspension and should be stirred continuously during use. A 10% solution of stannous chloride can be substituted for stannous sulfate.

5.5 Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in Type II water and dilute to 100 ml. Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.

5.6 Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 ml of Type II water.

5.7 Mercury stock solution: Dissolve 0.1354 g of mercuric chloride in 75 ml of distilled water. Add 10 ml of conc. nitric acid and adjust the volume to 100.0 ml (1.0 ml = 1.0 mg Hg).

5.8 Mercury working standard: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 µg/ml. This working standard and the dilution of the stock mercury solutions should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask as needed before adding the aliquot.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

7.0 Procedure

7.1 Sample preparation: Weigh triplicate 0.2-g portions of dry sample and place in the bottom of a BOD bottle. Add 5 ml of Type II water and 5 ml of aqua regia. Heat 2 min in a water bath at 95° C. Cool, add 50 ml Type II water and 15 ml potassium permanganate solution to each sample bottle. Mix thoroughly and place in the water bath for 30 min at 95° C. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. Add 55 ml of Type II water. Treating each bottle individually, add 5 ml of stannous sulfate and immediately attach the bottle to the aeration apparatus. Continue as described under 7.4.

7.2 An alternate digestion procedure employing an autoclave may also be used. In this method, 5 ml of conc. H_2SO_4 and 2 ml of conc. HNO_3 are added to the 0.2 g of sample. Add 5 ml of saturated $KMnO_4$ solution and cover the bottle with a piece of aluminum foil. The samples are autoclaved at 121° C and 15 lb for 15 min. Cool, dilute to a volume of 100 ml with Type II water and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Purge the dead air space and continue as described under 7.4.

7.3 Standard preparation: Transfer 0.0-, 0.5-, 1.0-, 2.0-, 5.0-, and 10-ml aliquots of the mercury working standard containing 0 to 1.0 µg of mercury to a series of 300-ml BOD bottles. Add enough Type II water to each bottle to make a total volume of 10 ml. Add 5 ml of aqua regia and heat 2 min in a water bath at 95° C. Allow the sample to cool and add 50 ml Type II water and 15 ml of $KMnO_4$ solution to each bottle and return to the water bath for 30 min. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Add 50 ml of Type II water. Treating each bottle individually, add 5 ml of stannous sulfate solution and immediately attach to bottle to the aeration apparatus and continue as described in Section 7.4.

7.4 Analysis: At this point, the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 liter/min, is allowed to run continuously. The absorbance, as exhibited either on the spectrophotometer or the recorder, will increase and reach maximum within 30 sec. As soon as the recorder pen

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levels off (approximately 1 min), open the bypass valve and continue the aeration until the absorbance returns to its minimum value. Close the bypass value, remove the fritted tubing from the BOD bottle, and continue the aeration.

7.5 Construct a calibration curve by plotting the absorbance of standards versus micrograms of mercury. Determine the peak height of the unknown from the chart and read the mercury value from the standard curve.

7.6 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.7 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.8 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7740

SELENIUM (ATOMIC ABSORPTION, FURNACE METHOD)

1.0 Scope and Application

1.1 Method 7740 is an atomic absorption procedure approved for determining the concentration of selenium in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7740, samples must be prepared in order to convert organic forms of selenium to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to the acid digestion procedure described in this method. Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is placed manually or by means of an automatic sampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. The absorption of hollow cathode radiation during atomization will be proportional to the selenium concentration.

2.3 The typical detection limit for this method is 2 µg/l.

3.0 Interferences

3.1 Elemental selenium and many of its compounds are volatile and therefore samples may be subject to losses of selenium during sample preparation. Spike samples and relevant standard reference materials should be processed to determine if the chosen dissolution method is appropriate.

3.2 Likewise, caution must be employed during the selection of temperature and times for the dry and char (ash) cycles. A nickel nitrate solution must be added to all digestate prior to analysis to minimize volatilization losses during drying and ashing.

3.3 In addition to the normal interferences experienced during graphite furnace analysis, selenium analysis can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Selenium analysis is particularly susceptible to these problems because of its low analytical wavelength (196.0 nm). Simultaneous background correction must be employed to avoid erroneously high results.

3.4 If the analyte is not completely volatilized and removed from the furnace during atomization, memory effects will occur. If this situation is detected, the tube should be cleaned by operating the furnace at full power at regular intervals in the analytical scheme.

3.5 Selenium analysis suffers interference from chlorides (more than 800 mg/l) and sulfate (more than 200 mg/l). The addition of nickel nitrate such that the final concentration is 1% nickel will lessen this interference.

4.0 Apparatus and Materials

4.1 250-ml Griffin beaker.

4.2 10-ml volumetric flasks.

4.3 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for simultaneous background correction and interfacing with a strip chart recorder.

4.4 Selenium hollow cathode lamp or electrodeless discharge lamp.

4.5 Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

4.6 Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can easily be recognized.

4.7 Pipets: Microliter with disposable tips. Sizes can range from 5 to 1000 µl as required.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Hydrogen peroxide (30%): Oxidant should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.4 Selenium standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 0.3453 g of selenious acid (actual assay 94.6% H_2SeO_3, analytical reagent grade) or equivalent in Type II water and dilute to 200 ml.

5.5 Nickel nitrate solution (5%): Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \cdot 6H_2O$ or equivalent in Type II water and dilute to 100 ml.

5.6 Nickel nitrate solution (1%): Dilute 20 ml of the 5% nickel nitrate to 100 ml with Type II water.

5.7 Selenium working standards: Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Withdraw appropriate aliquots of the stock solution, add 1 ml of conc. HNO_3, 2 ml of 30% H_2O_2, and 2 ml of the 5% nickel nitrate solution. Dilute to 100 ml with Type II water.

5.8 Air: Cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or a cylinder of industrial-grade compressed air.

5.9 Hydrogen: Suitable for instrumental analysis.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Special containers (e.g., containers used for volatile organic analysis) may have to be used if very volatile selenium compounds are to be analyzed.

6.4 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.5 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared in the manner described in Sections 7.1.1 to 7.1.3. Sludge-type samples should be prepared according to Method 3050, and samples containing oils, greases, or waxes may be prepared according to Methods 3030 or 3040. The applicability

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of a sample-preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.1.1 Transfer 100 ml of well-mixed sample to a 250-ml Griffin beaker, add 2 ml of 30% H_2O_2 and sufficient conc. HNO_3 to result in an acid concentration of 1% (v/v). Heat for 1 hr at 95° C or until the volume is slightly less than 50 ml.

7.1.2 Cool and bring back to 50 ml with Type II water.

7.1.3 Pipet 5 ml of this digested solution into a 10-ml volumetric flask, add 1 ml of the 1% nickel nitrate solution and dilute to 10 ml with Type II water. The sample is now ready for injection into the furnace.

7.2 The 196.0-nm wavelength line and a background correction system must be employed. Follow the manufacturer's suggestions for all other instrument parameters.

7.3 Furnace parameters suggested by the manufacturer should be employed as guidelines. Since temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

7.4 Inject a measured µl aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

7.5 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.6 Run a check standard after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced.

7.7 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.8 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors

must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7741

SELENIUM (ATOMIC ABSORPTION, GASEOUS HYDRIDE)

1.0 Scope and Application

1.1 Method 7741 is an atomic absorption procedure which is approved for determining the concentration of selenium in wastes, mobility procedure extracts, soils, and groundwater, provided that the sample matrix does not contain high concentrations of chromium, copper, mercury, silver, cobalt or molybdenum. All samples must be subjected to an appropriate dissolution step prior to analysis. Spiked samples and relevant standard reference materials are employed to determine applicability of the method to a given waste.

2.0 Summary of Method

2.1 Samples are prepared according to the nitric/sulfuric acid digestion procedure described in this method. Next, the selenium in the digestate is reduced to the +4 form using tin chloride. The +4 selenium is then converted to a volatile hydride with hydrogen produced from a zinc/HCl reaction.

2.2 The volatile hydride is swept into an argon-hydrogen flame located in the optical path of an atomic absorption spectrophotometer, and the resulting absorbance is proportional to the selenium concentration.

2.3 The typical detection limit for this method is 0.002 mg/l.

3.0 Interferences

3.1 High concentrations of chromium, cobalt, copper, mercury, molybdenum, nickel, and silver can cause analytical interferences.

3.2 Traces of nitric acid left following the sample workup can result in analytical interferences. Nitric acid must be distilled off by heating the sample until fumes of SO_3 are observed.

3.3 Elemental selenium and many of its compounds are volatile and therefore certain samples may be subject to losses of selenium during sample preparation.

4.0 Apparatus and Materials

4.1 100-ml beaker.

4.2 Electric hot plate.

4.3 A commercially available zinc slurry hydride generator or a generator constructed from the following material (see Figure 1).

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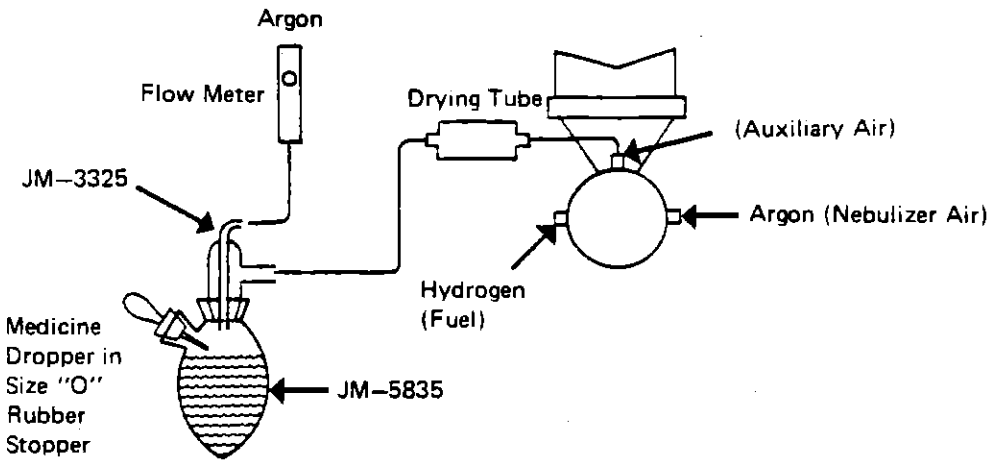

Figure 1. Zinc slurry hydride generator apparatus set-up and AAS sample introduction system.

4.3.1 Medicine dropper fitted into a size "0" rubber stopper capable of delivering 1.5 ml.

4.3.2 A 50-ml pear-shaped reaction flask with two 14/20 necks (Scientific Glass JM-5835).

4.3.3 Gas inlet-outlet tube constructed from a micro cold-finger condenser (JM-3325) by cutting the portion below the 14/20 ground-glass joint.

4.3.4 Magnetic stirrer to homogenize the zinc slurry.

4.3.5 A 10-cm polyethylene drying tube filled with glass to prevent particulate matter from entering the burner.

4.3.6 Flow meter capable of measuring 1 liter/min.

4.4 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for interfacing with a strip chart recorder and simultaneous background correction.

4.5 Burner recommended by the particular instrument manufacturer for the argon-hydrogen flame.

4.6 Selenium hollow cathode lamp or electrodeless discharge lamp.

4.7 Strip chart recorder.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Concentrated sulfuric acid: Acid should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.4 Concentrated hydrochloric acid: Acid should be analyzed to determine levels of impurities. If impurities are detected all analyses should be blank-corrected.

5.5 Diluent: Add 100 ml 18 N H_2SO_4 and 400 ml concentrated HCl to 400 ml Type II water and dilute to a final volume of 1 liter with Type II water.

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5.6 Potassium iodide solution: Dissolve 20 g KI in 100 ml Type II water.

5.7 Stannous chloride solution: Dissolve 100 g $SnCl_2$ in 100 ml of conc. HCl.

5.8 Selenium standard stock solution: 1000 mg/liter solution may be purchased, or prepared as follows. Dissolve 0.3453 g of selenious acid (assay 94.6% of H_2SeO_3) in Type II water. Add to a 200-ml volumetric flask and bring to volume (1 ml = 1 mg Se).

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Special containers (e.g., containers used for volatile organic analysis) may have to be used if very volatile selenium compounds are to be analyzed.

6.4 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.5 Nonaqueous samples shall be refrigerated where possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation

7.1.1 To a 50-g sample (or in the case of EP extracts a 50-ml sample) add 10 ml conc. HNO_3 and 12 ml of 18 N H_2SO_4. Evaporate the sample on a hot plate until white SO_3 fumes are observed (a volume of about 20 ml). Do not let it char. If it chars, stop the digestion, cool and add additional HNO_3. Maintain an excess of HNO_3 (evidence of brown fumes) and do not let the solution darken, because selenium may be reduced and lost. When the sample remains colorless or straw yellow during evolution of SO_3 fumes, the digestion is complete.

7.1.2 Cool the sample, add about 25 ml distilled deionized water and again evaporate to SO_3 fumes just to expel oxides of nitrogen. Cool. Add 40 ml conc. HCl and bring to a volume of 100 ml with distilled deionized water.

7.2 Prepare working standards from the standard stock solutions. The following procedure provides standards in the optimum working range.

7.2.1 Pipet 1 ml stock solution into a 1-liter volumetric flask. Bring to volume with Type II water containing 1.5 ml conc. HNO_3/liter. The concentration of this solution is 1 mg Se/liter (1 ml = 1 µg Se).

7.2.2 Prepare six working standards by transferring 0, 0.5, 1.0, 1.5, 2.0 and 2.5 ml of the selenium stock standard (see Section 5.8) into a 100-ml volumetric flasks. Bring to volume with diluent. The concentrations of these working standards are 0, 5, 10, 15, 20 and 25 µg Se/liter.

7.3 Standard additions

7.3.1 Take the 15-, 20-, and 25-µg standards and transfer quantitatively 25 ml from each into separate 50-ml volumetric flasks. Add 10 ml of the prepared sample to each. Bring to volume with Type II water containing 1.5 ml HNO_3/liter.

7.3.2 Add 10 ml of prepared sample to a 50-ml volumetric flask. Bring to volume with Type II water containing 1.5 ml HNO_3 per liter. This is the blank.

7.4 Follow the manufacturer's instructions for operating an argon-hydrogen flame. The argon-hydrogen flame is colorless so it may be useful to aspirate a low concentration of sodium to ensure that ignition has occurred.

7.5 The 196.0-nm wavelength shall be used for the analysis of selenium.

7.6 Transfer a 25-ml portion of the digested sample or standard to the reaction vessel. Add 0.5 ml $SnCl_2$ solution. Allow at least 10 min for the metal to be reduced to its lowest oxidation state. Attach the reaction vessel to the special gas inlet-outlet glassware. Fill the medicine dropper with 1.50 ml zinc slurry that has been kept in suspension with the magnetic stirrer. Firmly insert the stopper containing the medicine dropper into the side neck of the reaction vessel. Squeeze the bulb to introduce the zinc slurry into the sample or standard solution. The metal hydride will produce a peak almost immediately. When the recorder pen returns partway to the base line, remove the reaction vessel.

7.7 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.8 Duplicates, spiked samples, and check standards should be routinely analyzed.

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7.9 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. For example, if the method of standard additions was employed, the analytical value will be one-tenth the concentration of the original sample due to dilution during preparation. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7760

SILVER (ATOMIC ABSORPTION, DIRECT ASPIRATION METHOD)

1.0 Scope and Application

1.1 Method 7760 is an atomic absorption procedure approved for determining the concentration of silver in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7760, samples must be prepared for direct aspiration. The method of sample preparation will vary according to the sample matrix. Aqueous samples are subjected to the acid digestion procedure described in this method. Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is aspirated into an air/acetylene flame. The resulting absorption of hollow cathode radiation will be proportional to the silver concentration. Background correction must be employed for all analyses.

2.3 The typical detection limit for this method is 0.01 mg/l; typical sensitivity is 0.06 mg/l.

3.0 Interferences

3.1 Background correction should be employed since nonspecific absorption and light scattering may occur at the analytical wavelength.

3.2 Silver nitrate solutions are light-sensitive and have the tendency to plate out on container walls. Thus silver standards should be stored in brown bottles.

3.3 Silver chloride is insoluble so hydrochloric acid should be avoided unless the silver is already in solution as a chloride complex.

3.4 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument, having a grating monochromator, photomultiplier detector, adjustable slits, and provisions for background correction.

4.2 Silver hollow cathode lamp.

4.3 Strip chart recorder (optional).

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Concentrated ammonium hydroxide (NH_4OH): Base should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.4 Silver standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products or Fisher Scientific) and verify by comparison with a second standard, or dissolve 0.7874 g anhydrous silver nitrate ($AgNO_3$), analytical reagent grade, in Type II water. Add 5 ml conc. HNO_3 and bring to volume in a 500-ml volumetric flask (1 ml = 1 mg Ag).

5.5 Silver working standards: These standards should be prepared with nitric acid and at the same concentrations as the analytical solution.

5.6 Iodine solution, IN: Dissolve 20 g potassium iodide (KI), analytical reagent grade, in 50 ml Type II water. Add 12.7 g iodine (I_2), analytical reagent grade, and dilute to 100 ml. Place in a brown bottle.

5.7 Cyanogen iodide solution: To 50 ml deionized distilled water add 4.0 ml conc. NH_4OH, 6.5 g KCN, and 5.0 ml of iodine solution. Mix and dilute to 100 ml with deionized distilled water. Do not keep longer than 2 weeks. CAUTION: This reagent cannot be mixed with any acid solutions since toxic hydrogen cyanide will be produced.

5.8 Air: Cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or a cylinder of industrial-grade compressed air.

5.9 Acetylene: Should be of high purity. Acetone, which is usually present in acetylene cylinders, can be prevented from entering and affecting flame conditions by replacing the cylinder before the pressure has fallen to 50 psig.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 When possible standards and samples should be stored in the dark and in brown bottles.

6.5 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared according to Sections 7.2 and 7.3; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 Preparation of aqueous samples

7.2.1 Transfer a representative aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of conc. HNO_3. Cover the beaker with a watch glass. Place the beaker on a hot plate and cautiously evaporate to near dryness, making certain that the sample does not boil. (DO NOT BAKE.) Cool the beaker and add another 3-ml portion of conc. HNO_3. Re-cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs.

7.2.2 Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Again, evaporate to near dryness and cool the beaker. Add a small quantity of HNO_3 so that the final dilution contains 0.5% (v/v) HNO_3, and warm the beaker to dissolve any precipitate or residue resulting from evaporation.

7.2.3 Wash down the beaker walls and watch glass with distilled water and, when necessary, filter the sample to remove silicates and other insoluble material that could clog the nebulizer. Adjust the

volume to some predetermined value based on the expected metal concentrations. The sample is now ready for analysis.

7.3 If plating out of AgCl is suspected, the precipitate can be redissolved by adding cyanogen iodide to the sample. CAUTION: This can only be done after digestion to prevent formation of toxic hydrogen cyanide under acid conditions. If cyanogen iodide addition to the sample is necessary, then the standards must be treated in the same manner. CAUTION: cyanogen iodide must not be added to the acidified silver standards. New standards must be made as directed in Sections 5.4 and 5.5 except that the acid addition step must be omitted. Transfer 10 ml of stock solution to a small beaker. Add Type II water to make about 80 ml. Make the solution basic (pH above 7) with ammonium hydroxide. Rinse the pH meter electrodes into the solution with Type II water. Add 1 ml cyanogen iodide and allow to stand 1 hr. Transfer quantitatively to a 100 ml volumetric flask and bring to volume with Type II water.

7.4 The 328.1-nm wavelength line and background correction shall be employed.

7.5 An oxidizing air/acetylene flame shall be used.

7.6 Follow the manufacturer's operating instructions for all other spectrophotometer parameters.

7.7 Either (1) run a series of silver standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.8 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.9 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.10 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

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METHOD 7761

SILVER (ATOMIC ABSORPTION, FURNACE METHOD)

1.0 Scope and Application

1.1 Method 7761 is an atomic absorption procedure approved for determining the concentration of silver in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 Summary of Method

2.1 Prior to analysis by Method 7761, samples must be prepared in order to convert organic forms of silver to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to the acid digestion procedure described in this method. Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is placed manually or by means of an automatic sampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. The absorption of hollow cathode radiation during atomization will be proportional to the silver concentration.

2.3 The typical detection limit for this method is 0.2 µg/l.

3.0 Interferences

3.1 The long residence time and high concentrations of the atomized sample in the optical path of the graphite furnace can result in severe physical and chemical interferences. Furnace parameters must be optimized to minimize these effects.

3.2 In addition to the normal interferences experienced during graphite furnace analysis, silver analysis can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Simultaneous background correction must be employed to avoid erroneously high results.

3.3 If the analyte is not completely volatilized and removed from the furnace during atomization, memory effects will occur. If this situation is detected, the tube should be cleaned by operating the furnace at higher atomization temperatures.

3.4 Silver nitrate solutions are light sensitive and have the tendency to plate out on container walls. Thus silver standards should be stored in brown bottles.

4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for simultaneous background correction and interfacing with a strip chart recorder.

4.2 Silver hollow cathode lamp.

4.3 Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

4.4 Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can easily be recognized.

5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Lead standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products or Fisher Scientific) and verify by comparison with a second standard, or dissolve 0.7874 g anhydrous silver nitrate ($AgNO_3$), analytical reagent grade, in Type II water. Add 5 ml concentrated HNO_3 and bring to volume in a 500-ml volumetric flask.

5.4 Silver working standards: These standards should be be prepared with nitric acid such that the final acid concentration is 0.5% (v/v) HNO_3.

5.5 Concentrated ammonium hydroxide (NH_4OH): Base should be analyzed to determine levels of impurities. If impurities are detected, all analyses should be blank-corrected.

5.6 Iodine solution (IN): Dissolve 20 g potassium iodide (KI), analytical reagent grade, in 50 ml Type II water. Add 12.7 g iodine (I_2), analytical reagent grade, and dilute to 100 ml. Place in a brown bottle.

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5.7 Cyanogen iodide solution: To 50 ml deionized distilled water add 4.0 ml concentrated NH_4OH, 6.5 g KCN, and 5.0 ml of iodine solution. Mix and dilute to 100 ml with deionized distilled water. Do not keep longer than 2 weeks. CAUTION: This reagent cannot be mixed with any acid solutions since highly toxic hydrogen cyanide will be produced.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared according to Section 7.2; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 Preparation of aqueous samples

7.2.1 Transfer a representative aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of conc. HNO_3. Cover the beaker with a watch glass. Place the beaker on a hot plate and cautiously evaporate to near dryness, making certain that the sample does not boil. (DO NOT BAKE.) Cool the beaker and add another 3-ml portion of conc. HNO_3. Re-cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs.

7.2.1 Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Again, evaporate to near dryness and cool the beaker. Add a small quantity of HNO_3 so that the final dilution contains 0.5% (v/v) HNO_3, and warm the beaker to dissolve any precipitate or residue resulting from evaporation.

7.2.3 Wash down the beaker walls and watch glass with distilled water and, when necessary, filter the sample to remove silicates and other insoluble material that could clog the nebulizer. Adjust the volume to some predetermined value based on the expected metal concentrations. The sample is now ready for analysis.

7.3 If plating out of AgCl is suspected, the precipitate can be redissolved by adding cyanogen iodide to the sample. CAUTION: This can only be done after digestion to prevent formation of toxic hydrogen cyanide under acid conditions. If cyanogen iodide addition to the sample is necessary, then the standards must be treated in the same manner. CAUTION: cyanogen iodide must not be added to the acidified silver standards. New standards must be made as directed in Sections 5.4 and 5.5 except that the acid addition step must be omitted. Transfer 10 ml of stock solution to a small beaker. Add Type II water to make about 80 ml. Make the solution basic (pH above 7) with ammonium hydroxide. Rinse the pH meter electrodes into the solution with Type II water. Add 1 ml cyanogen iodide and allow to stand 1 hr. Transfer quantitatively to a 100-ml volumetric flask and bring to volume with Type II water.

7.4 The 328.1-nm wavelength line and background correction shall be used.

7.5 Follow the manufacturer's operating instructions for all other spectrophotometer parameters.

7.6 Furnace parameters suggested by the manufacturer should be employed as guidelines. Since temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

7.7 Inject a measured µl aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

7.8 Either (1) run a series of silver standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.9 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.10 Run a check standard after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced.

7.11 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.12 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

8.0 Quality Control

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

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

SECTION D

PROCESS INFORMATION

This section discusses process information for the storage of drummed wastes and wastes in the tank. Ashland will store drummed wastes until a truckload quantity is accumulated for shipment to a permitted disposal site. Wastes will be accumulated in the tank until the tank is nearly full and then shipped in bulk for disposal.

D-1 Containers

D-1a Containers With Free Liquids

The wastes which are to be stored at this facility are listed in the Part A application. The chemical and physical characteristics of these wastes are described in the waste analysis plan (Section C). These hazardous wastes will contain free liquids. Therefore, the storage area is designed for drums containing free liquids.

The hazardous waste storage area is located outdoors north of the R&D lab. Hazardous wastes have been stored in this area in accordance with interim status standards for waste containers (40 CFR Part 265 Subpart I, Use And Management Of Containers). This storage area will be upgraded to meet the final standards for waste containers as specified in 40 CFR Part 264 Subpart I.

This will require the construction of a ramp and curbing around the waste drum storage area. A layout of the drum storage area showing construction details and spacing of drums is shown on the drawing in the Appendix. A compliance schedule for construction of the storage ramp and curbing is shown in Table D-1.

The container storage pad covers an area of 8,753 ft^2. Of this area approximately 3,147 ft^2 will be used for storage of hazardous waste. There will be 888 ft^n for the ramp areas providing forklift access to the storage area. There will be 1,559 ft^2 of aisle space and the remaining 700 ft^n will be actual storage area.

The storage area is divided into seven sections which are separated by curbs to permit the storage of incompatible wastes. At the present time the majority of the wastes we store are compatible. However, since we are a research facility the nature of these wastes could change at any time. This design will give us maximum flexibility in waste storage.

Stacked two drums high the four storage areas on the west side have the capacity to hold as many as 40 drums or a total of 80 drums each. The three storage areas on the east side have the capacity to hold as many as 20 drums or a total of 40 drums each. The maximum number of hazardous waste drums which are expected to be in storage at any one time are 400 drums. Due to the parameters used to design this storage area and the fact that we currently have only a few drums of incompatible wastes, the maximum designed drum storage is 440. This will allow us to store nearly 400 drums in the first 6 bays and store the few remaining drums of incompatible waste in the 7th bay.

The basis for determining which wastes are to be stored in a particular bay are the known chemical and physical properties of the wastes. This information is obtained from the Ashland Chemical R&D Lab's internal chemical waste profile sheet which each generator in the lab must complete. Since these internal forms are completed by chemists or chemical technicians, no further analyses are necessary to characterize each waste. At the present time the principle source of incompatibility is between corrosives (D002) and the chemicals with which they may react.

D-1a(1) Description Of Containers

The containers which are used for a particular waste must meet DOT regulations for that waste. The container which meets the DOT regulations for the waste is compatible with the waste stored in it. During weekly inspections of the storage area any leaking drum caused by incompatibility of the waste and drum would be noticed. The contents of the drum would be transferred to another more compatible drum if the leaking were caused by waste/drum incompatibility. The containers which Ashland uses for the different hazardous wastes are described in the Standard Practice Instructions K-17 which are attached.

Ashland personnel who are responsible for filling, shipping and receiving hazardous waste containers are trained to check the specification markings on the containers to see that they comply with DOT regulations.

D-1a(2) Container Management Practices

Waste containers are always kept closed when in storage. This facility serves only as a storage facility for wastes generated in Ashland research and development operations.

The waste container storage area is inspected weekly to ensure that the containers are stored in a manner to prevent ruptures and leaks. The containers are stored on wooden pallets. The storage area is checked to determine that the pallets and containers are properly placed on the pallets.

The pallets are adequately spaced for inspection. The maximum number of drums expected to be in storage at any one time are 400 drums.

The container storage area is outside and away from sources of ignition. The storage area is 380 feet from the property line. Drums of incompatible wastes are stored in separate bays. No self-reactive or explosive wastes are permitted on site.

Drums and pallets are moved and handled with special forklift trucks.

D-1a(3) Secondary Containment System Design & Operation

The containment system design is shown in the drawing in the Appendix. The storage pad is constructed of 6 inch thick concrete. A 9 inch high curb will surround three sides of the storage area. The entrance ramp will be sloped up from base grade to curb height and then back down to the storage pad floor.

The storage pad area for waste storage will be divided into seven storage bays by six internal curbs of 9 inch height. The large storage bays will hold 10 pallets as shown. The small storage bays will hold 5 pallets. A total of 80 55-gallon drums can be stored in the larger bays and 40 55-gallon drums can be stored in the smaller bays when pallets are stacked 2 high.

The capacity of the secondary containment system is calculated as follows:

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Cell #1

1. The far western bay of the storage area has the dimensions of 12'9" x 28' and a curb height of 9 inches. Contained volume is therefore:

   $12.75 \text{ ft} \times 28 \text{ ft} \times \frac{9}{12} \text{ ft} = 267.8 \text{ ft}^3 \times 7.48 \text{ gal/ft}^3 = 2003$ gallons

2. Volume of ramp:

   $\frac{9}{12} \text{ ft} \times 12.75 \text{ ft} \times 5.5 \text{ ft} \times (\frac{1}{2}) (7.48 \text{ gal/ft}^3) = 197$ gallons

3. Volume of pallets used:

   $V = 10 \text{ pallets } (2.6 \text{ ft}^3)/\text{pallet } (7.48 \text{ gal/ft}^3) = 194$ gallons

4. Volume of drums stored:

   $V = (1)^2 \text{ft}^2 (.750 - .469)\text{ft} \times 40 \times 7.48 \text{ gal/ft}^3 = 264$ gallons

5. Net available spill containment volume:

   $= 2003 + 197 - 194 - 264 = 1742$ gallons

6. Required volume = 10% of amount stored + 24 hour 100 year rainfall = .10 (80 drums x 55 gals/drum) + (4.56 in x ft/12 in) (12.75 ft) (33.5 ft) x 7.48 gals/ft^3 = 440 + 1214 = 1654 gallons

Cells 2, 3, & 4

1. The 2, 3, and 4 bays from the west of the storage area have the dimensions of 12' 6" x 28' and a curb height of 9 inches. Contained volume is therefore:

   $12.5 \text{ ft} \times 28 \text{ ft} \times \frac{9}{12} \text{ ft} = 262.5 \text{ ft}^3 \times 7.48 \text{ gal/ft}^3 = 1964$ gallons

2. Volume of ramp $\frac{9}{12}$ ft x 12.5 ft x 5.5 ft x ($\frac{1}{2}$)(7.48 gal/ft^3) = 193 gallons

3. Volume of pallets used: Same as Cell #1 194 gallons

4. Volume of drums stored: Same as Cell #1 264 gallons

5. Net available spill containment volume:

   $= 1964 + 193 - 194 - 264 = 1699$ gallons

6. Required volume = .10(80 drums x 55 gals/drum) + (4.56in x $\frac{\text{ft}}{12\text{in}}$)(12.5ft)(33.5ft) x 7.48 gals/ft^3 = 440 + 1190 = 1630 gallons

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Cells 5 & 6

1. The 5 and 6 bay from the west of the storage area have the dimensions of 8' 6" x 28' and a curb height of 9 inches. Contained volume is therefore:

   $8.5 \text{ ft} \times 38 \text{ ft} \times \frac{9}{12} \text{ ft} = 178.5 \text{ ft}^3 \times 7.48 \text{ gal/ft}^3 = 1335 \text{ gallons}$

2. Volume of ramp:

   $\frac{9}{12} \text{ ft} \times 8.5 \text{ ft } 5.5 \text{ ft} \times (\tfrac{1}{2})(7.48 \text{ gal/ft}^3) = 131 \text{ gallons}$

3. Volume of pallets used:

   $V = 5 \text{ pallets } (2.6 \text{ ft}^3/\text{pallet})(7.48 \text{ gal/ft}^3) = 97 \text{ gallons}$

4. Volume of drums stroed:

   $V = (1)^2 \text{ ft}^2(.750 - .469)\text{ft} \times 20 \times 7.48 \text{ gal/ft}^3 = 132 \text{ gallons}$

5. Net available spill containment volume:

   $1335 + 131 - 97 - 132 = 1237 \text{ gallons}$

6. Required volume = $.10\ (40 \text{ drums} \times 55 \text{ gals/dr}) + (4.56 \text{ in.} \times \frac{\text{ft}}{12\text{in}})(8.5 \text{ ft})(33.5 \text{ ft}) \times 7.48 \text{ gal/ft}^3 = 220 + 809 = 1029 \text{ gallons}$

Cell 7

1. The cast bay has the dimensions of 9'7" x 28' and a curb height of 9 inches. Contained volume is therefore:

   $9.6 \text{ ft} \times 28 \text{ ft} \times \frac{9}{12} \text{ ft} = 201.6 \text{ ft}^3 \times 7.48 \text{ gal/ft}^3 = 1508 \text{ gallons}$

2. Volume of ramp:

   $\frac{9}{12} \text{ ft} \times 9.6 \text{ ft} \times 5.5 \text{ ft} \times (\tfrac{1}{2})(7.48 \text{ gal/ft}^3) = 148 \text{ gallons}$

3. Volume of pallets used: Same as Cells 5 & 6 97 gallons

4. Volume of drums stored: Same as Cells 5 & 6 132 gallons

5. Net available spill containment volume:

   $1508 + 148 - 97 - 132 = 1427 \text{ gallons}$

6. Required volume = $.10\ (40 \text{ drums} \times 55 \text{ gallons/dr}) + (4.56 \text{ in.} \times \frac{\text{ft}}{12\text{in}})(9.6 \text{ ft})(33.5 \text{ ft}) \times 7.48 \text{ gals/ft}^3 = 220 + 914 = 1134 \text{ gallons}$

D-1a(3)(a) Requirement For The Base To Contain Liquids

The storage pad base is 6 inch thick concrete. This base will be sufficiently impervious to contain leaks and spills. Any leak or spill will be cleaned up immediately.

D-1a(3)(b) Containment System Drainage

The waste drums are stored on pallets to protect the bottoms from contact with spilled liquids. The pallets elevate the drums by 5.6 inches.

D-1a(3)(c) Containment System Capacity

Wastes are stored in 55-gallon drums. The maximum number of drums that will be stored in a storage bay are 80 drums (4400 gallons). Each larger storage bay has secondary containment for 1742 gallons for bay 1 and 1699 gallons for bays 2, 3 and 4. Each smaller bay has secondary containment for 1237 gallons for bays 5 and 6 and 1427 gallons for bay 7.

D-1a(3)(d) Control Of Run-On

Run-on is prevented from entering the containment area by 9 inch curb around the storage area.

D-1a(4) Removal Of Spilled Liquids

The R&D Lab has a spill control plan for removing spilled liquids. Any liquid spill or leak will be cleaned up immediately. Small spills will be neutralized and/or absorbed with material kept in inventory for such purposes. Large spills will be picked up with a portable pump.

All spills will be transferred to drums that are compatible with the spilled material and that meet DOT requirements for hazardous wastes. The spilled material will be sampled and submitted for approval for disposal at a permitted disposal site.

The lack of a roof may result in some accumulation of rainwater during heavy storms. The secondary containment system has adequate capacity to hold this water in addition to 10% of the stored waste.

The storage area will be checked following a heavy rainfall to see if any rain-on has occurred. Any rainwater accumulation will be drummed off. A sample of the rainwater will be analyzed to determine if it has been contaminated. The sample will be analyzed for any organic carbon using a gas chromatograph and a pH will be run. If the organic carbon content is significant, a TOC will be run to further characterize the carbon.

If pH is in the range of 6.5-8.5 and TOC, if run, are at background level the rainwater will be discharged to the city sewer. If the rainwater is contaminated a sample will be submitted for approval for disposal at a permitted disposal site.

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Table D-1
COMPLIANCE SCHEDULE FOR WASTE CONTAINER STORAGE AREA

DUBLIN

Activity	Days after effective date of Permit							
	20	40	60	80	100	120	140	160
1. Request For Contract Bids	→							
2. Award Bid & Prepare Contract		→						
3. Order Materials, Prepare Forms			→					
4. Placement Of Concrete & Curing				→	→			
5. Certification Of Construction & Submittal To Ohio EPA						→		

D-2 Tank

The wastes which are stored in the tank are listed in the Part A application. The chemical and physical characteristics of this waste are described in the waste analysis plan (Section C).

The spent solvent storage tank is located west of the R&D Dock. Hazardous wastes have been stored in this tank in accordance with interim status standards for tanks (40 CFR Part 265 Subpart J - Tanks).

D-2a Description of Tank

An 8,000 gallon underground tank provides storage for wastes generated from cleaning operations in the pilot plant in which reactors and other miscellaneous equipment are rinsed to remove resins. The wash solvent would be a mixture of the following solvents: toluene, xylene, methyl ethyl ketone, ethyl acetate and possibly other ignitable solvents which are both listed and not listed.

The tank is 316 stainless steel and is 21'0" long and 8'0" in diameter. The attached drawing gives additional information. The shell of the tank is ¼ inch thick. The tank has the following nozzles/openings:

1. A 4 inch schedule 10 316 stainless steel draw pipe. This draw pipe goes to within 4 inches of the bottom of the tank and rises 3'6" above the top of the tank. There is a 150# carbon steel flange at the top of the draw pipe connecting it to the line which is used to empty the tank into a tanker.

2. A 3 inch vent which is approximately 6 inches above the top of the tank. This vent has a 150# 316 stainless steel flange on it which is attached to a vent pipe coming up through the ground.

3. A 20 inch manway which is bolted with an asbestos gasket.

4. A 3 inch spare vent which is identical to the other vent except it is blind flanged.

5. A 2 inch schedule 10 316 stainless steel fill pipe. The fill pipe goes within 6 inches of the bottom of the tank.

The tank is buried outside the R&D Laboratory near the R&D Loading Dock. When the tank was installed a concrete slab was poured under the tank to anchor the tank.

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D-2b Tank Corrosion and Erosion

The tank is 316 stainless steel for chemical and corrosion resistance. Stainless steel tanks have been shown to be very resistant to a variety of organic solvents. The table on the following page lists all F001, F002, F003 and F005 solvents which we are obtaining a permit to store in the underground tank. A review of this table shows the majority of these solvents have an average penetration rate of less than .002 inches per year. The highest corrosion rate is .02 inches per year. These values are given at 77°F which is the lowest temperature given in this source. Therefore, the corrosion rate will be lower at the underground temperature of the tank. At the given range of corrosion rates, the expected life of the tank was calculated using an average rate of .011 for the less than .020 rate since the rate is between .002 and .020. A value of .001 was used for the less than .002 rate since the rate is between .002 and .0. The expected life of the tank would be 23 to 250 years.

At the time of installation in 1980 the tank was pressure tested at between 3-5 psi and was found to be satisfactory.

During normal operations the gauge is checked approximately once a week when material is pumped into the tank. If there is any significant discrepancy in the level of solvents in the tank, an engineer in the Engineering Plant Services Group in the Administration Building will be consulted. Further testing will be done based upon his recommendations.

D-2c Tank Management Practices

A simplified process flow diagram is shown of the following page. Basically the tank is a holding tank for spent wash solvents from the pilot plant. The pilot plant uses solvents such as toluene, xylene, methyl ethyl ketone, ethyl acetate and various other F listed solvents and unlisted ignitable solvents to clean reactors and other equipment. The spent products are collected in drums in the pilot plant and transported to the solvent storage room for temporary storage prior to pumping into the waste storage tank. A pump is used to transfer the waste from the drum to the waste storage tank. Prior to transfer the continuous purge manometer tank gauge (Meriam Instrument Model 30 FP25 WM) is read and recorded on the tank log. It there is a significant discrepancy from the last reading, the Plant Services Group in the Engineering Department of Ashland Chemical will be consulted to determine what additional testing should be done to insure the integrity of the tank. When the tank is nearly full, the spent solvents are pumped to a tanker and transporter to an incinerator for disposal.

Other than the gauge, there is no instrumentation on this tank. There is no waste feed cutoff system since the wastes are manually pumped into the tank. There would be no possibility of major loss in overfilling the tank since the operator pumping the waste from the drums to the tank is in constant attendance during the transfer process.

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Corrosion Rate for F001, F002, F003 and F005 Wastes
on 316 Stainless Steel

Corrosive	Average Penetration Per Year Inches at 77°F
Tetrachloroethylene	<.002
Trichloroethylene	<.002
Methylene Chloride	<.002
1,1,1 Trichloroethane	<.002
Carbon Tetrachloride	<.002
Chlorinated Fluorocarbons	Generic Class - Not Listed
Chlorobenzene	<.002
1,1,2-Trichloro 1,2,2-Trifluoroethane	<.002
Ortho-dichlorobenzene	<.020
Trichlorofluoromethane	Not Listed
Xylene	<.020
Acetone	<.002
Ethyl Acetate	<.002
Ethyl Benzene	<.020
Ethyl Ether	<.002
Methyl Isobutyl Ketone	<.020
n-Butyl Alcohol	<.020
Cyclohexanone	<.020
Methanol	<.002
Toluene	<.002
Methyl Ethyl Ketone	<.002
Carbon Disulfide	<.020
Isobutanol	Not Listed
Pyridine	<.002

Source: Corrosion Data Survey, Metals Section, 5th Edition, Complied by Norman Hamner, National Association of Corrosion Engineers.

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There is a 3 inch atmospheric vent in the tank. Since the tank is underground emissions from this vent would occur only during filling.

The tank level indicator is checked approximately weekly. The tank is filled approximately once a year which means that approximately three drums are pumped into the tank each week. At this low level of activity checking prior to loading is adequate.

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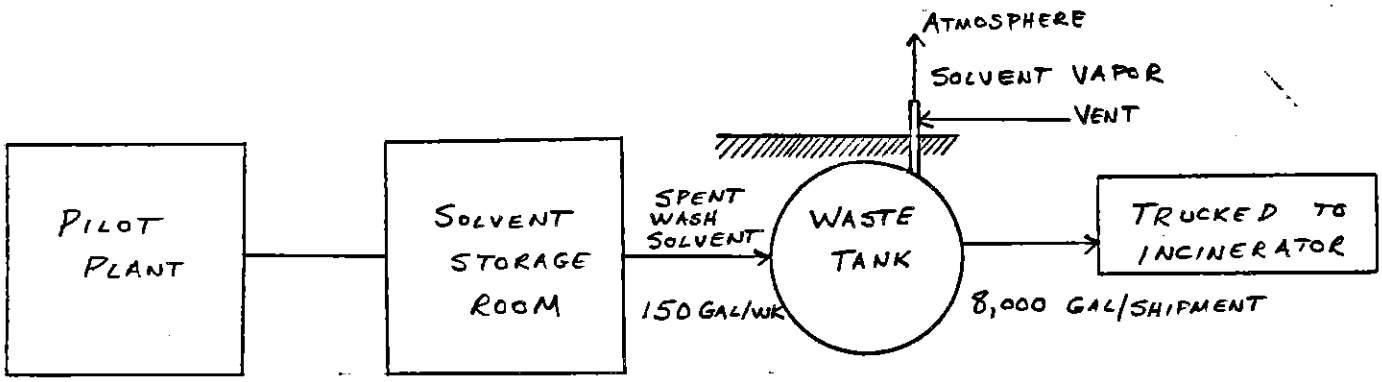
427B-AC

CALCULATION SHEET

ASHLAND CHEMICAL COMPANY, ENGINEERING DEPARTMENT

DATE: 2/5/86

SUBJECT: DUBLIN R+D WASTE TANK - PROCESS FLOW DIAGRAM			SHEET NO. 1	
			AFE NO.	
BY: AA HENDRICKSON	CHECKED BY:	APPROVED BY:	ENGR. NO. 6020	DRAWING NO. 1



WASTE GENERATION SITE	TEMPORARY DRUM STORAGE OF WASTE	BULK STORAGE OF WASTE	BULK SHIPMENT OF WASTE

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D-7.6

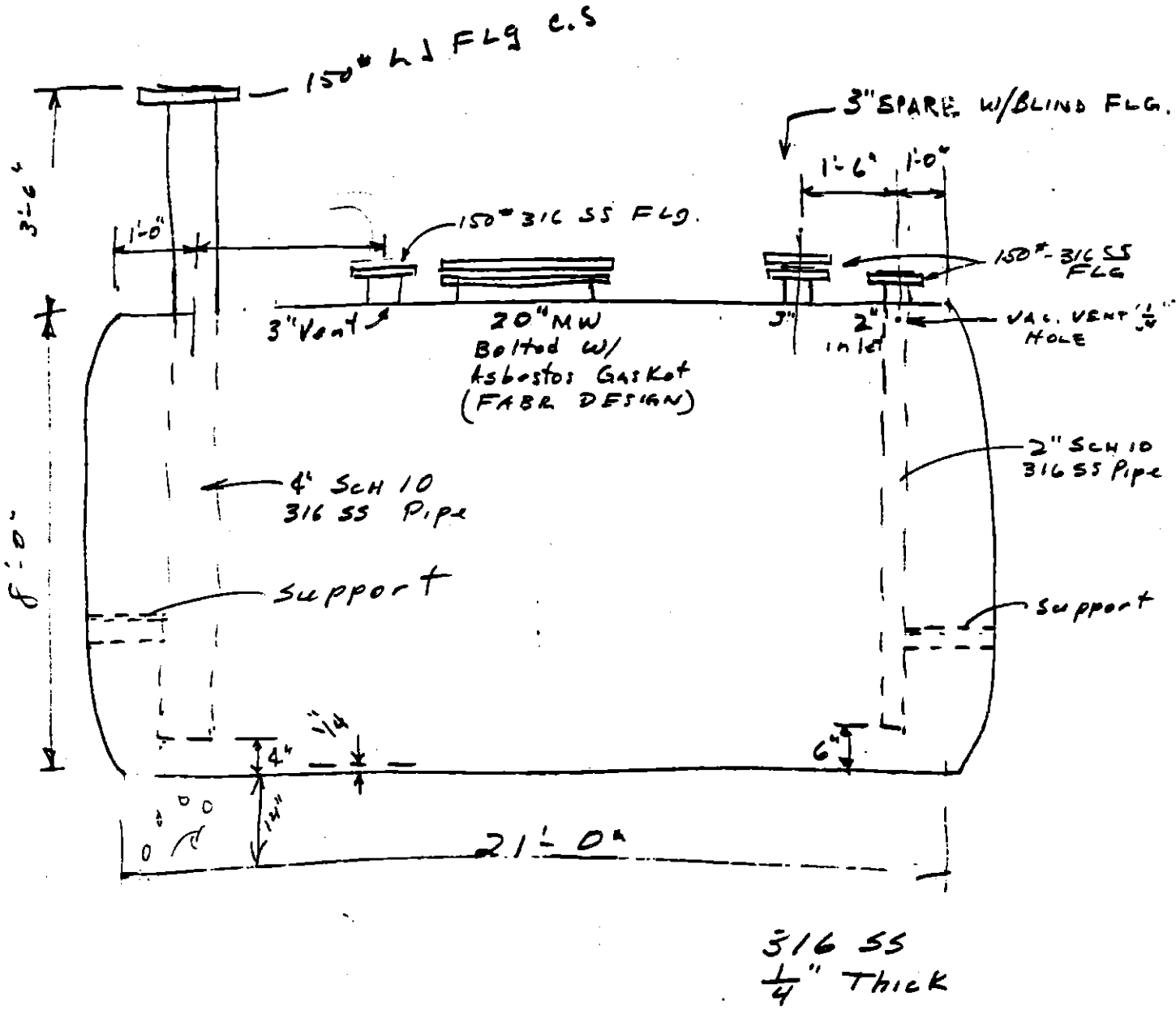
LATION SHEET

ND CHEMICAL COMPANY, ENGINEERING DEPARTMENT

DATE: 10-30-79

CT: SCRAP SOLVENT STORAGE TANK - RESEARCH LAB

BY: | CHECKED BY: | APPROVED BY: | ENG. NO. E-3172 | DRAWING NO



316 SS
1/4" Thick

JOB # 80606
RECEIVED
1008
CODE
SIGNED

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P-8

STANDARD PRACTICE INSTRUCTIONS

DEPARTMENT	INSTRUCTION NO.	PREPARED BY	APPROVED		DATE PREVIOUS INSTRUCTIONS	DATE ISSUED
IC&S Division	K-17	PHR			11-18-73	3-26-75

SUBJECT: CONTAINERS FOR HAZARDOUS CHEMICALS

D.O.T. regulations governing the shipment of hazardous materials must be strictly obeyed by all IC&S plants. The regulations cover shipment by land, water, or air, by any type of public or private carrier and apply to any destination.

Part of these regulations cover specifications covering legal shipping containers for materials classified by D.O.T. as hazardous.

IC&S is legally responsible for the use of authorized containers for any hazardous chemicals shipped from its plants by its own truck or other carrier, regardless of whether the container was filled in the plant or by a supplier. However, a "shipper may accept the manufacturer's certification or specification marking to determine that containers were manufactured in accordance with applicable specifications." This means that IC&S district personnel who are responsible for filling and delivery of any hazardous material by its truck or public carrier, must know what packages are authorized for every such product and check the specification markings on the containers to be sure they comply with regulations.

The following categories are classified by D.O.T. as hazardous materials:

Flammable liquid
Flammable solid
Non flammable gas
Poison - Class B
Oxidizing material
Corrosive material

It should be noted that the grouping of products for a particular D.O.T. requirement, such as packaging shown above, does not necessarily mean the same group of products are subject to a different D.O.T. requirement, such as labeling.

Flammable liquids are currently defined by D.O.T. as those with a Tag closed cup flash point below 100° F. Most IC&S flammable products fall into one of two groups:

Those with a T.C.C. flash point of 20°F or below and a Reid vapor pressure not over 16 p.s.i., absolute, at 100°F.

Those with a T.C.C. flash point above 20°F to 100°F and vapor pressure not over 16 p.s.i., absolute, at 100°F.

Containers authorized by D.O.T. for use with products in both these groups are similar except that when the flash point is 20°F or below, drum openings must not exceed 2.3 inches in diameter. All containers must be of "a design and constructed of materials that will not react dangerously with or be decomposed by the chemical packed therein". This includes any lining or liners used. If this is not known, assurance should be obtained from the manufacturer of the drum, the vendor of the product, or our Manager of Laboratories and Quality Control.

The most frequently used 55 gallon drum authorized for flammable solvents are Spec 17C and 17E. The 17C is constructed of 16 gauge steel, both body and head sheet. We often use 17C drums lined with an epoxy phenolic resin to reduce the possibility of contamination of the product. Care must be taken not to ship any solvents in such lined drum that would react with or dissolve any lining.

The 17E drum is constructed of 18 gauge steel, both body and head. If the flash point is above 20°F, 17E drums constructed of 20 gauge body sheet and 18 gauge head are authorized. However, this means it is illegal to use 20 gauge body with 18 gauge head 17E drums for such solvents as Hexane, Rubber Solvent, Hexasol, Cyclohexane, Acetone, T.H.F., or any other with flash point 20°F or below. If it is not practical to use all 17E drums of 18 gauge body and head sheet, these must be carefully separated at the District. Be very careful to use only 17E drums made entirely of 18 gauge steel for any product with a T.C.C. flash point of 20°F or less.

Both 17C and 17E are single trip containers and, therefore, are not reusable drums in the sense of simply rinsing and/or refilling for hazardous materials. The regulations do permit reuse of some single trip containers for flammable liquids, including the 17C and 17E, if certain conditions are met which include thorough cleaning, inspection, and testing of each package to be reused. Each drum must be in such condition, including closures, that it complies in all respects with the requirements for these containers. A special permit from the D.O.T. for this reconditioning is required. Such permits, if desired, should be obtained through our Traffic Department in Dublin. Drums can also be purchased from outside suppliers who are authorized by D.O.T. permits to recondition such drums. When such drums are received, be sure the permit number is stencilled on the drum. (See SPI K-8.)

For returnable drums not requiring complete reconditioning and testing after each trip, Spec 5, 5A, and 5B are authorized. Removable heads are authorized for the Spec 5 and 5B, but not for 5A.

Smaller packages than the usual 55 gallon drums are also subject to the regulations, such as 5 gallon and 1 gallon packages. The same specification containers, such as 17C and 17E, can be used, and the gauge thickness required is less for smaller containers. For example, 24 gauge for capacity not over 5 gallons. Metal cans not over 1 gallon each may also be shipped in Spec 12B fiberboard boxes, but they cannot be shipped without this overpack.

For liquids with a vapor pressure which exceeds 16 p.s.i., absolute, at 100°F, but does not exceed 27 p.s.i., absolute, at 100°F, (includes ethyamine, 70-72%, propylamine, isopropylamine, and pentane and any other products with vapor pressure over 16 p.s.i.), all drums previously described are authorized except 17E, which are not permitted.

STANDARD PRACTICE INSTRUCTIONS

DEPARTMENT	INSTRUCTION NO.	PREPARED BY	APPROVED		DATE PREVIOUS INSTRUCTIONS	DATE ISSUED
IC&S Division	K-17	PHR			2-5-73	11-18-73

SUBJECT: CONTAINERS FOR HAZARDOUS CHEMICALS

Corrosive material is the next most frequent group of IC&S products subject to D.O.T. packaging regulations. Several of the corrosive products sold by the Division, such as muriatic, nitric, and sulfuric acids, are specifically covered by name in the regulations. Others fall into more general categories, such as "corrosive liquids not specifically provided for".

Authorized containers for corrosive liquids fall into two groups: reusable and non-reusable. D.O.T. regulations do not permit the reconditioning and reuse of single trip containers for corrosive liquids.

Examples of "alkaline corrosive liquids n.o.s. (not otherwise specified)" are caustic soda liquid and caustic potash liquid. Examples of "corrosive liquids not specifically provided for" are acetic acid and phosphoric acid. The main returnable containers for this group of products are polyethylene or polyethylene containers in wood or steel outer containers. Others are often purchased in supplier packages.

"Corrosive solids not specifically provided for" are normally sold as packaged by the supplier of the product. It can be assumed these are properly selected, but every container should be marked with a specification number. Corrosive solids cannot be repackaged by a District, unless there is assurance that the container meets specification requirements from an authority such as the Traffic Department in Dublin. Examples of this group of products are caustic soda and caustic potash (flake, granular, etc.), sodium sulfhydrate, sodium sulfide, sodium tetrasulfide, ammonium bifluoride, etc.

Flammable solids and oxidizing materials are normally sold in the packages furnished by the supplier and cannot be repackaged by a District. Examples of oxidizing materials are: chromic acid, sodium nitrite, sodium nitrate, potassium nitrate, sodium chlorate, ammonium bichromate, sodium hydrosulfite, and calcium hypochlorite.

Poisons (Class B) are similarly regulated and sold in the original package. Typical poisons include all the cyanides, several plating salts, brighteners, and addition agents, aniline and phenol.

Compressed gasses are either sold in cylinders provided by the supplier or filled under conditions established by our operations and engineering departments. These requirements are so specialized that any such requirements should be first discussed with the Operations Manager.

Containers for blends should be obtained from the Blend Manual or from the Manager of Laboratories and Quality Control, so that authorized packages are used.

Temporary special permits have been issued by the Department of Transportation to some companies, including Ashland Chemical Company, authorizing shipment of certain corrosive liquids in 55 gallon reusable molded polyethylene containers for use without overpack, made in compliance with Spec. 34 except for prescribed provisions. These permits authorize shipment and reuse of any product for which Spec. 34 reusable polyethylene containers are authorized.

Special Permit	Manufacturer	Expiration Date
DOT E 6637	Advanced Chemical Technology	February 5, 1978
DOT E 6700	Container Corporation of America	February 20, 1978

Use of containers under this special permit carries certain restrictions which must be complied with, as follows:

1. Every container must be marked with the D.O.T. special permit number.

2. All shipping papers covering shipments using any such containers must be marked with D.O.T. special permit number opposite the listing of the product shipped in them. This includes shipment by either common or privite carrier.

3. A report must be made to the Traffic Department in Dublin whenever any material is lost in the course of storage or shipment of product in these drums. The report should include an explanation of the circumstances causing the loss.

4. Purchase of containers believed to conform to this special permit should be done exclusively through the Purchasing Department of Ashland Chemical Company in Dublin.

Products which are often packaged in such returnable containers by IC&S Districts include:

Acetic acid - all concentrations
Formic acid - all concentrations
Gluconia acid - all concentrations
Hydrofluosilicic acid - all concentrations
Muriatic acid - all concentrations
Phosphoric acid - 75%, 80%, or 85%
Sulfuric acid - up to and including 66° (1.835 Sp. Gr.)
Caustic Soda - up to and including 50%
Caustic Potash - up to and including 45%
Battery acid based on sulfuric acid - all concentrations up to 1.835 Sp. Gr.
Cleaning Compound, liquid - based on above commodities
Sodium Hupochlorite solution - containgng not over 7% available chlorine by weight
Hydrogen Peroxide solution - containing not over 52% hydrogen peroxide by weight

STANDARD PRACTICE INSTRUCTIONS

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IC&S Division	K-17	PHR			2-5-73	11-18-73

SUBJECT: CONTAINERS FOR HAZARDOUS CHEMICALS

Tank truck shipments are also covered as a part of the D.O.T. packaging regulations. A tank truck must have specification numbers that indicate it is authorized for the hazardous material to be shipped and the material of construction to be in contact with the product must be a type approved for that product.

Every bulk shipment from an IC&S district must be in an approved cargo tank, both as to specification number and material of construction. This applies regardless of the owner of the equipment. When tank trucks are provided by another carrier, the regulations authorize the shipper (Ashland) to accept certification signed by an authorized agent of the carrier that the cargo tank is a proper container for the shipment. The wording for this is:

> "Carrier certifies that the cargo tank supplied for this shipment is a proper container for the transportation of this commodity as described by shipper.
> Carrier _____
> By _____"

This certification is printed on the bills of lading provided by some carriers. For cases where it is not, or if an Ashland bill of lading is used, a copy of the papers which will be retained must be stamped with these words, using a rubber stamp provided to each IC&S District. In any case, this must be signed by an authorized agent of any public carrier to insure that our company is blameless in case an unauthorized tank should be used.

If there is any question as to whether a product is classified as hazardous, consult the Freight Classifications and Hazardous Designations manual which has been supplied to each IC&S District by the Traffic Department in Dublin.

If there is any question as to whether a given container is authorized, consult the Traffic Department in Dublin.

The subject of Containers for Hazardous Chemicals is also covered in Audio-Visual Program No. 08.

STANDARD PRACTICE INSTRUCTIONS

DEPARTMENT	INSTRUCTION NO.	PREPARED BY	APPROVED		DATE PREVIOUS INSTRUCTIONS	DATE ISSUED
IC&S Division	K-17	PHR			2-5-73	11-18-73

SUBJECT: CONTAINERS FOR HAZARDOUS CHEMICALS

Muriatic Acid - Returnable Containers

Spec 1H metal crate with inside polyethylene carboy of Spec 2T, capacity 13 gallons.

Spec 16D wirebound wooden overwrap with inside Spec 2T, 2TL, 2S or 2 SL polyethylene container, capacity 13 gallons

Spec 6D steel drum with Spec 2S or 2SL inside polyethylene container, capacity up to 55 gallons.

Spec 60 portable tank may be used; specification rubber lining is required; no bottom outlets are permitted.

Spec 34 polyethylene container without overwrap, such as the Del drum; maximum capacity on this specification is 30 gallons. Temporary permits have been issued by D.O.T. to Ashland authorizing shipment of certain corrosive liquids in 55 gallon reusable molded polyethylene containers.

Spec 1A, 1C, or 1K wood box with glass carboy.

Muriatic Acid - Non Reusable Containers

Spec 37M steel drum with Spec 2S or 2 SL polyethylene inside container.

Spec 21P fiber drum overpack with 2S, 2 SL, or 2U polyethylene inner container.

For 1 gallon containers, Spec 12A, or 12B fiberboard boxes with inside polyethylene containers not over 1 gallon capacity each are authorized. Containers must be cushioned to prevent movement in the case and gross weight of the completed package must not exceed 65 pounds.

Sulfuric Acid 66° (or 1.835 electrolyte grade) - Returnable Containers

Spec 1H metal crate with inside Spec 2T polyethylene carboy, capacity 13 gallons.

Spec 16D wirebound wooden overwrap with Spec 2T, 2TL, 2S, or 2SL polyethylene container, up to 13 gallons.

Spec 6D steel drum with Spec 2S or 2SL inner polyethylene container. The steel drum must be minimum 16 gauge steel throughout. (The use of only minimum 16 gauge Spec 6D steel drums with Spec 2S or 2SL inner container would make the use possible for all corrosive liquids normally repackaged except Nitric Acid.)

Spec 34 polyethylene container, such as Del drum, capacity not over 30 gallons. Temporary permits required for 55 gallon capacity.

Sulfuric Acid - Returnable Containers (continued)

Spec 60 portable tanks may be used for sulfuric acid 66°. Steel is satisfactory for technical grade; no bottom outlets are permitted.

Spec 1A, 1C, or 1K box with glass carboy.

Sulfuric Acid - Non Reusable Containers

Spec 12P fiberboard boxes with inside Spec 2U polyethylene containers not over 5 gallons. Wire staples are not permitted to close the boxes. Spec 12B fiberboard boxes to contain 4 inside polyethylene containers not over 1 gallon capacity, suitably cushioned to prevent movement. For any other non-returnable packages, consult the Traffic Department in Dublin.

Nitric Acid - Returnable Containers

Spec 5C stainless steel drums, capacity 15 or 55 gallons. No drums should be used for nitric acid unless they are embossed with this Spec 5C; and no container weighing less than 85% of its original marked weight may be used.

Spec 1A, 1C, or 1K straight sided carboys in boxes or kegs; with cushioning for carboys of approved incombustible material.

Spec 60 portable tanks of approved construction; consult Traffic Department for details.

Hydrofluoric Acid - Returnable Containers

Spec 6D steel overpack with 2S or 2SL polyethylene inner container.

Spec 16D wirebound wooden overwrap with inside Spec 2T polyethylene container.

Hydrofluoric Acid - Non Reusable Containers

Spec 37M steel overpack with 2S, 2SL, or 2T polyethylene containers. Any overpack over 15 gallon capacity must be constructed entirely of at least 20 gauge steel.

Hydrofluosilicic Acid (not exceeding 32% concentration) - Returnable Containers

Spec 6D cylindrical steel overpack with 2S or 2SL polyethylene inner container.

Spec 34 polyethylene container, such as Del drum, not exceeding 30 gallon capacity.

Hydrofluosilicic Acid (not exceeding 32% concentration) - Non Reusable Containers

Spec 37M steel overpack with inside 2S or 2SL container, except the overpack must be constructed of at least 20 gauge steel and not exceed 16 gallon capacity.

Spec 21P fiber drum overpack with inside 2S, 2SL, or 2U polyethylene inner container.

Hydrogen Peroxide (not exceeding 52% concentration) - Returnable Containers

Spec 42D aluminum drums with vented closure in top head; maximum capacity of 55 gallons. Top heads must be clearly marked "Keep This End Up" or "Keep Plug Up To Prevent Spillage".

Spec 6D steel overpack with 2S or 2SL inside polyethylene containers, with closures vented and end marked as for the 42D drums.

Spec 34 polyethylene containers, not over 30 gallon capacity, similarly vented and marked.

Hydrogen Peroxide (not exceeding 52% concentration - Non Reusable Containers

Spec 21P fiber drum overpack with inside Spec 2SL polyethylene container, with 30 gallon capacity; or Spec 2U container, with 15 gallon capacity. Closures must be vented and head with the closure marked "Keep This End Up" or "Keep Plug Up To Prevent Spillage".

Hypochlorite Solutions (not exceeding 16% concentration) - Returnable Containers

Spec 34 polyethylene (Del drum).

Spec 1H metal crate outer container with Spec 2T polyethylene inner container.

Spec 15P glued plywood or wooden box with Spec 2S polyethylene inner container.

Spec 6D cylindrical steel overpack, with 2S or 2SL polyethylene inner container; 55 gallon capacity.

Hypochlorite Solutions (not exceeding 16% concentration) - Non Reusable Containers

Spec 37M cylindrical steel overpack with inside 2S or 2SL polyethylene liner.

Spec 12P fiberboard box with inside Spec 2U polyethylene inner container, capacity not over 5 gallons. Wire staples must not be used to close this outer container.

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STANDARD PRACTICE INSTRUCTIONS

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IC&S Division	K-17	PHR			2-5-73	11-18-73

SUBJECT: CONTAINERS FOR HAZARDOUS CHEMICALS

Caustic Soda Liquid or Caustic Potash Liquid - Returnable Containers

Spec 5 or 5A steel drum.

Spec 6D steel drum with 2S or 2SL liner.

Spec 60 portable steel tank.

Spec 34 polyethylene container (Del drum), capacity not over 30 gallons. Temporary permits required for 55 gallon capacity.

Caustic Soda Liquid or Caustic Potash Liquid - Non Reusable Containers

Spec 17C, 17E, or 17F steel drums. These drums cannot be reconditioned and reused for corrosives.

Acetic Acid or Phosphoric Acid - Returnable Containers

Spec 6D steel drum with 2S or 2SL polyethylene inner container, with 55 gallon capacity.

Spec 16A wirebound wooden box with 2U polyethylene inner container.

Spec 34 polyethylene container (Del drum), capacity not over 30 gallons. Temporary permits required for 55 gallon capacity.

Spec 5C stainless steel drum. For Phosphoric Acid, Spec 5C, 316 alloy stainless steel.

Acetic Acid or Phosphoric Acid - Non Reusable Containers

Spec 37M steel overpack with Spec 2S or 2SL inner liner.

Spec 37P steel drum with polyethylene liner for materials that will not react with polyethylene and result in container failure.

Spec 21P fiber drum overpack with inside Spec 2S, 2SL, or 2U polyethylene container.

Cargo Tank Shipments

PRODUCT	D.O.T. APPROVED SPEC. AND MATERIAL OF CONSTRUCTION
Flammable or other non-corrosive liquids with vapor pressure less than 3 p.s.i. gauge, trichlorethylene, perchlorethylene, and Norgechlor. Liquids over 3 p.s.i. gauge vapor pressure may be shipped in MC-306 but MC-306 is vented at this pressure	MC-306 or MC-307 steel, stainless steel, or aluminum
Flammable or other non-corrosive liquids with vapor pressure or unloading pressure not over 25 p.s.i. gauge	MC-307 steel, stainless steel, or aluminum
But for methylene chloride, 1,1,1 trichlorethane, or blends containing chlorinated solvents with other solvents such as aliphatics	MC-306 or MC-307 depending on vapor pressure as previously described. Steel or stainless steel. Do not use aluminum without permission from the Traffic Department.
For liquids where the vapor pressure is over 25 p.s.i. gauge or unloading pressure is to exceed 25 p.s.i.	MC-330 steel, stainless steel, or aluminum

Same limitations on products containing chlorinated solvents as previously mentioned.

Plants operating MC-300 or other coded tanks up to MC-306 or 307 are permitted to continue their use except for the limitations described for chlorinated solvents or blends.

For Corrosive materials, Sulfuric Acid 66°Tech	MC-310 or MC-312 steel or Heresite lined steel. No bottom outlets for acids
For Sulfuric Acid 1.835 Electrolyte Grade	MC-310, MC-311, or MC-312 steel lined with Heresite (to preserve the low iron content)
Nitric Acid (any concentration)	MC-310, MC-311, or MC-312 stainless steel
Muriatic Acid (any concentration)	MC-310, MC-311, or MC-312 steel, rubber lined in accordance with specs.
Phosphotic Acid (any concentration)	MC-310, MC-311, or MC-312 stainless steel or steel, rubber lined

SECTION E

GROUNDWATER MONITORING SYSTEMS

These requirements apply to facilities that treat, store, or dispose of hazardous wastes in surface impoundments, waste piles, land treatment units or landfills. They therefore do not apply to this facility.

SECTION F

PROCEDURES TO PREVENT HAZARDS

F-1 Security

The entire drum storage pad is enclosed by a 6 foot 9½ inch high chain link fence. Access to the pad is through two gates. One gate is at the southwest corner and the other gate is at the northeast corner. The Northeast gate is locked at all times unless the well pump is being serviced.

The main gate is shut and locked on the weekend and evenings when the lab is not in operation.

Two warning signs are in place at the gate and on the fence surrounding the pad. One sign says, "Danger - Unauthorized Personnel Keep Out". The other warning sign says, "Danger - No Smoking, Matches or Open Lights". The locations of these signs are indicated on the drawing of the Hazardous Waste Drum Storage Area in the Appendix.

The underground solvent storage tank is located to the north of the solvent storage room in the R&D Building. The fill line for this tank is in the solvent storage room. Access to this room is through one door. This door is open only during normal working hours when personnel are present. The door is locked on the weekend and evenings.

F-2 Inspection Schedule

The Resource Conservation and Recovery Act and Oil Pollution Prevention Regulations (SPCC Plan) require that a facility is routinely inspected for conditions that may be causing or may pose a threat to human health.

The facility must have a written inspection schedule. The inspection schedule must identify the types of problems to look for during the inspection.

Inspections must be recorded in a log. The log must be kept for three years from the date of inspection. Deteriorations or malfunctions revealed by the inspection must be remedied before they lead to an environmental or human health hazard.

The inspection records must include the following minimum information:

(1) The date of inspection

(2) The time of inspection

(3) The name of the inspector

(4) A notation of the observations made

(5) The date and nature of any repairs or other remedial actions

F-2a General Inspection Requirements

Laboratory facilities are inspected routinely to insure safe working conditions. Laboratory chemists and technicians are responsible for daily visual inspection of the areas under their supervision. This includes inspection of tanks, pipes, fittings, pumps and hoses to insure proper working order. Incoming trucks are visually inspected. Each chemist is responsible for checking the emergency equipment in his area daily to make sure it is intact.

Laboratory emergency and safety equipment which are related to hazardous waste storage are inspected monthly. The loading area on the drum storage pad near the southwest gate is inspected daily when loading is being done. These inspections are recorded in inspection logs. The monthly inspection logs for emergency equipment are attached. The inspection logs identify the problems that are to be looked for during inspection.

In addition to monthly inspections of emergency equipment, monthly safety meetings are conducted. Employees are trained in the use of emergency equipment such as fire extinguishers and breathing gear. Employees are trained in fire fighting, dealing with spills and in plant evacuation procedures.

Plant security devices which are related to hazardous waste storage are inspected weekly. This includes the storage pad fence, both gates, and the warning signs. A weekly inspection log is attached for the security devices. The log identifies the problems that are to be looked for during the inspection.

F-2b Inspection of the Waste Container Storage Area

The waste container storage area is inspected weekly. The weekly inspection log for the waste container storage area is attached. The log identifies the problems that are to be looked for during inspection.

The storage area is checked for proper housekeeping. The area is checked for cleanliness in general and for the proper placement and stacking of containers. The stacks are checked for damaged and leaking containers and damaged or broken pallets.

The containers are checked to see that they are in good condition, properly sealed, and properly coded. The drums are checked for loose bungs or open lids. The drum codes are checked to see that incompatible wastes are not being stored in the same area.

The base or floor of the storage area is checked for cracks, wet spots or erosion. The ramp to the storage area and the curbs are checked for cracks or settlement. The containment system is checked for leaks or liquid spills.

F-2c Inspection of the Underground Waste Solvent Tank

The underground waste solvent tank is inspected whenever solvents are pumped into the tank. This has not been regularly scheduled, but records indicate that this has been done approximately on a weekly basis. In the future, the tank will be inspected each operating day or at least once a week depending on which is more frequent. The inspection log for the underground waste solvent tank is attached. The log identifies problems that are to be checked during an inspection.

The pipes, valves and fittings that are visable aboveground are checked for signs of leaks, corrosion or deterioration. The tank guage is checked by comparing the present reading with the final reading the last time material was transferred into the tank. If the readings are not consistant, a manual reading will be made using a guage stick. The pump will be checked to insure that it is not clogged and has proper power. The ground above the tank outside will be checked for signs of wet spots or dead vegetation.

F-2d Remedial Action

As described on the Inspection Logs on pages F-3 to F-6 remedial action or repairs will be done before the problem results in damage to human health or the environment. In the loading area any cracks, erosion, wet spots, or settlement will be repaired before any potential spilled material could reach the soil under the asphalt. Dirty floors and trash in the area will be cleaned up before it results in an accident. Any spill or leak will be cleaned up immediately and the spill residue will be considered a hazardous waste and disposed of properly.

In the container storage area the container placement and stacking will be changed if it does not meet the standard. Open lids or loose bungs will be closed or tightened immediately upon inspection. Contents of containers which show signs of corrosion, leakage or are defective will be placed into empty drums. Damaged pallets will be replaced with good pallets. Any cracks, erosion, wet spots or settlement in the concrete base, ramps or curbs will be repaired before any potential spilled material could reach the soil under the concrete pad. Dirty floor and trash in the area will be cleaned up before an accident. Warning signs which are illegible or damaged will be replaced as soon as new signs can be ordered. A liquid spill or leak will be cleaned up immediately and the spill residue will be disposed of properly.

On the tank, any signs of leaks, corrosion or deterioration of pipes, valves, fittings, etc., will be repaired before any potential spilled material reaches the soil under the equipment. Appropriate cleanup measures will be taken if there are signs of wet spots or dead vegetation on the soil. If there are problems with the tank gauge sticking or not giving consitant readings, a manual reading will be taken using a gauge stick. The contents of the tank are measured prior to and after pumping solvents into the tank. The accuracy of the gauge can be judged in this manner since the volume of waste pumped into the tanks is known. If there are problems with the pump, it will be repaired or another pump used.

During the security device inspection, any damage or corrosion to the chain link fence will be corrected if it is in such a location to compromise security. Likewise any corrosion or damage to the gates or locks will be corrected. Any missing warning signs will be replaced. Any damage or corrosion to the solvent storage room door/lock will be repaired.

The following emergency equipment will be inspected and replaced if absent or not present in sufficient quantity; absorbent material, sand supply, chemical neutralizers, fire extinguishers, first aid kit, various items of personal protective equipment listed and empty drums. The fork lift and telephones must be in working order and will be repaired if defective.

UM STORAGE PAD

ervations	Date & Type of Repairs/Action

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F-3

WEEKLY CONTAINER STORAGE AREA INSPECTION LOG

Date:_____
Time:_____
Inspector:_____

Item	Types Of Problems	Status O.K.	Status Not O.K.	Observations	Date & Type Of Repairs/Action
Container Placement & Stacking	Aisle Space, Height Of Stacks				
Sealing Of Containers	Open Lids Or Loose Bungs				
Labeling Of Containers	Improper Identification				
Containers	Corrosion, Leakage, Defective Drums				
Pallets	Damaged				
Base Or Foundation	Cracks, Erosion Wet Spots				
Housekeeping In Area	Dirty Floors, Trash In Area				
Ramps And Curbs	Settlement, Wet Spots				
Warning Signs	Readable, Damaged or Missing				
Containment System	Liquid Spill Or Leak				

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F-4

WEEKLY SECURITY DEVICES INSPECTION LOG SHEET

Inspector's Name:_____
Date Of Inspection:_____

Item	Types Of Problems	Status O.K.	Status Not O.K.	Observations	Date & Type Of Repair/Action
Drum Storage Pad	Corrosion, Damage To Chain Link Fence				
Main Gate - Southwest	Corrosion, Damage To Gate & Lock				
Northeast Gate	Corrosion, Damage To Gate				
Warning Signs	Are Signs Readable Damaged Missing				
Solvent Storage Room Door	Corrsoion, Damage To Lock				

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F-5

Date: _____
Time: _____
Inspector: _____

MONTHLY INSPECTION LOG FOR EMERGENCY EQUIPMENT

Item Inspected	Problems To Look For		Use Status O.K.	Use Status Not O.K.	Observations	Remedial Action
Absorben Material	Sufficient Quantity Bags/Bales In Good Condition					
Fork Lif	In Proper Operation Condition					
Sand Supply	Sufficient Quantity					
Chemical Neutralizers	Sufficient Quantity; Bags In Good Condition					
Telephone	In Working Condition					
1. Fire Extinguisher (17#)	Is Seal Intact	1				
2. Fire Extinguisher (17#)	Corrosion Of Case	2				
3. Fire Extinguisher (17#)	Damage To Hose	3				
4. Fire Extinguisher (17#)	Gauge Reading	4				
First Aid Kit (1st Floor R&D)	All Items Present					
Personnel Protective Clothing & Equipment (1st Floor R&D)						
1. Hard Hats & Goggles 2. Safety Shoes 3. Work Gloves	Sufficient Quantity					
1. Acid Master Suit 2. Acid Master Suit	Present And In Good Condition					
1. Scott Air Pak	In Work Condition	1				
2. Scott Air Pak	Check Tank Pressure	2				
3. Scott Air Pak	Gauge	3				
4. Scott Air Pak		4				
5. Scott Air Pak		5				
6. Scott Air Pak		6				
7. Scott Air Pak		7				
8. Scott Air Pak		8				
9. Scott Air Pak		9				
10. Scott Air Pak		10				
11. Scott Air Pak		11				
12. Scott Air Pak		12				
13. Scott Air Pak		13				
14. Scott Air Pak		14				
15. Scott Air Pak		15				
Empty Drums	In Good Condition					

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F-6

INSPECTION LOG FOR UNDERGROUND WASTE SOLVENT TANK

(Each Operating Day or Weekly, Whichever is More Frequent)

Date _____

Time _____

Inspector _____

Item	Types of Problems	Status OK	Status Not OK	Observations	Date & Type of Repairs/Action
Pipes, Valves, Fittings, Etc.	Leaks, Corrosion, Deterioration				
Tank Gauge	Sticking				
Pump	Clogging, Power				
Ground Around Pipes & Fittings	Wet Spots, Dead Vegetation				
Gauge Reading	Not Consistant With Last Reading				

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F-6.1

OR UNDERGROUND WASTE SOLVENT TANK

r Weekly, Whichever is More Frequent)

	Status		Observations	Date & Type of Repairs/Action
	OK	Not OK		
n				
ng				

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F-6.1

F-3 Preparedness & Prevention

F-3a Equipment Requirements

F-3a(1) Internal Communications

The drum storage area has a telephone approximately 40 feet from the main gate (see drawing in Appendix). This telephone is connected to the Ashland guard desk which is attended 24 hrs a day. The underground waste tank and fill line into the tank are located in the solvent storage room beside the R&D Dock where R&D personnel are in constant attendance. There is a phone on the adjacent dock to communicate with individuals in the lab or internal security.

F-3a(2) External Communications

The company phone system is used for summoning emergency assistance from the local police and fire departments and from state emergency response teams. The guard at the main desk would make the necessary calls for the drum storage pad. For the underground waste tank area, the phone on the adjacent dock can be used for outside calls to summon required emergency assistance.

F-3a(3) Emergency Equipment

The plant is equipped with emergency equipment to combat a fire, explosion or spill of product or hazardous waste.

Fire water supply is from the Ashland well. Fire water hoses and hydrants are located at strategic areas of the lab. In addition to the fire water supply, portable fire extinguishers are distributed throughout the lab. There are four fire extinguishers located on the fence surrounding the drum storage pad.

Scott air packs and air canisters are available for breathing protection. First aid supplies are maintained in the lab.

Chemical neutralizers and absorbents are available for combatting spills. Empty drums are available into which the contents of drums can be placed.

A listing of plant emergency equipment is contained in the plant contingency plan which is part of this permit application.

F-3a(4) Water For Fire Control

Fire water supply is available at fire plugs from a 128,000 gallon holding tank in which Ashland stores water. Water is available at 80 psi pressure to supply fire hoses for fire control. Fire hoses are also available.

F-3b Aisle Space Requirement

The waste drum storage area is inspected weekly for proper placement of containers and pallets. The storage area is maintained in good order with adequate aisle space for placement of pallets.

F-4 Preventive Procedures, Structure, & Equipment

F-4a Prevention of Hazards in Unloading/Loading Operations

Drums are stored, loaded and unloaded on wooden pallets. A forklift is used to load and unload pallets to and from trucks. Drums will be loaded onto trucks or pumped into tankers on the south end of the drum storage pad near the southwest gate. Any spill or leaking drum will be cleaned up immediately. The spill residue will be a hazardous waste and disposed of properly. At least one person will be in constant attendance during loading operations to insure that spills are cleaned up immediately.

F-4b Prevention of Runoff From Hazardous Waste Handling Areas to Other Areas of the Facility or Environment or Prevention of Flooding

The storage area will be curbed to prevent runoff to other areas. The storage pad is located outside of the 100 year floodplain. The curbing was designed to contain 10% of the volume stored in addition to a 24 hr. 100-year rainfall.

The solvent storage room is completely diked on 4 sides with a ramp at the entrance door. There is no floor drain in the room.

The solvent storage room where wastes are transferred from drums to the tank is indoors, therefore, there will be no runoff from this area to outside of the lab. The area is dock high and not in the 100-year flood zone. Therefore, a flood would not affect this area.

F-4c Prevention of Contamination of Water Supplies

The storage area will be surrounded by a 9 inch curb on 3 sides and 9 inch ramp on the 4th side. This was designed to handle leaks as well as rainfall.

Since an employee is in constant attendance during transfer operations into the tank, there is no danger of contamination during transfer operations. The tank is constructed of 316 stainless steel which offers the best corrosion resistance for material we store in the tank. The weekly inspection of the tank and guage reading determinations offer assurance that the tank is not leaking and prevents contamination of water supplies. Also, there is no floor drain in the solvent storage room. The solvent storage room is completely diked.

F-4d Mitigation of Effects of Equipment Failure and Power Outages

Operations in the hazardous waste storage area would not be effected by a power outage. The principal potential equipment failure would be damaged or leaking containers.

The contents of a damaged container would be placed in an empty drum. In the event of a leaking container the lab spill control plan would be implemented. The lab spill control plan is described in the Contingency Plan section of this permit application.

A spill would be contained. The leaked material would be treated with a neutralizer and/or absorbent. The absorbed spill would then be cleaned up.

Operations in the underground tank likewise would be unaffected by equipment failure or power outage. If the pump or gauge malfunctions, a backup pump or manual gauge will be used. The material can remain in drums for safe storage if transfer equipment is malfunctioning. During a power outage, transfer operations would cease.

F-4e Prevention of Undue Exposure of Personnel to Hazardous Waste

Ashland maintains standards for protective clothing and equipment that are required for the workplace. These standards are described in Research & Development Safety Manual. The Safety Manual describes the protective clothing and equipment that are required. It specifies the protective clothing and equipment that must be used in the lab.

All personnel exposed to possible contact from hazardous waste when sampling a drum must wear adequate body protection to prevent injury. The minimum clothing required are chemical resistant goggles, full length rubber apron, gauntlet gloves and rubber boots other safety shoes.

A copy of the R&D Safety Manual is attached to the Contingency Plan.

F-5 Prevention of Reaction of Ignitable, Reactive or Incompatible Waste

F-5a Precautions to Prevent Ignition or Reaction of Ignitable or Reactive Waste

The waste drums are stored in an area which is protected from accidental ignition sources. Smoking is not permitted in this area and "No Smoking" signs are conspicuously posted.

Hazardous wastes that may be potentially reactive with each other will be separated by curbing.

The entire solvent storage room is a non-smoking area with "No Smoking" signs conspicuously posted.

F-5b General Precautions For Handling Ignitable or Reactive Waste and Mixing of Incompatible Waste

No mixing of incompatible wastes is done by this facility.

F-5c Ignitable or Reactive Wastes in Containers

The waste container storage area is more than 50 feet from the property line (see the Site Plan and Topographical Survey).

The waste solvents which are stored in drums are temporarily stored in the pilot plant during accumulation or in the solvent storage room. Both areas are more than 50 feet from the property line.

F-5d Incompatible Wastes in Containers

Incompatible wastes are not put into the same container. No mixing of wastes is done on site. Unwashed containers which held incompatible wastes will not be used as transfer containers.

Incompatible wastes will be separated by curbing.

F-5e Ignitable or Reactive Wastes in Tanks

Ignitable wastes are stored in the underground storage tank. No reactive wastes are stored in the tank. The waste is stored so that it is protected from any material or conditions which may cuase the waste to ignite by having the tank underground and by having the fill line to the pipe in an area protected from sources of ignition such as open flames, smoking, cutting and welding, hot surfaces, frictional heat, sparks (static, electrical, or mechanical), spontaneous ignition and radiant heat. Smoking is confined to specially designated areas of the R&D Building. "No Smoking" signs are conspicuously placed around the solvent storage room.

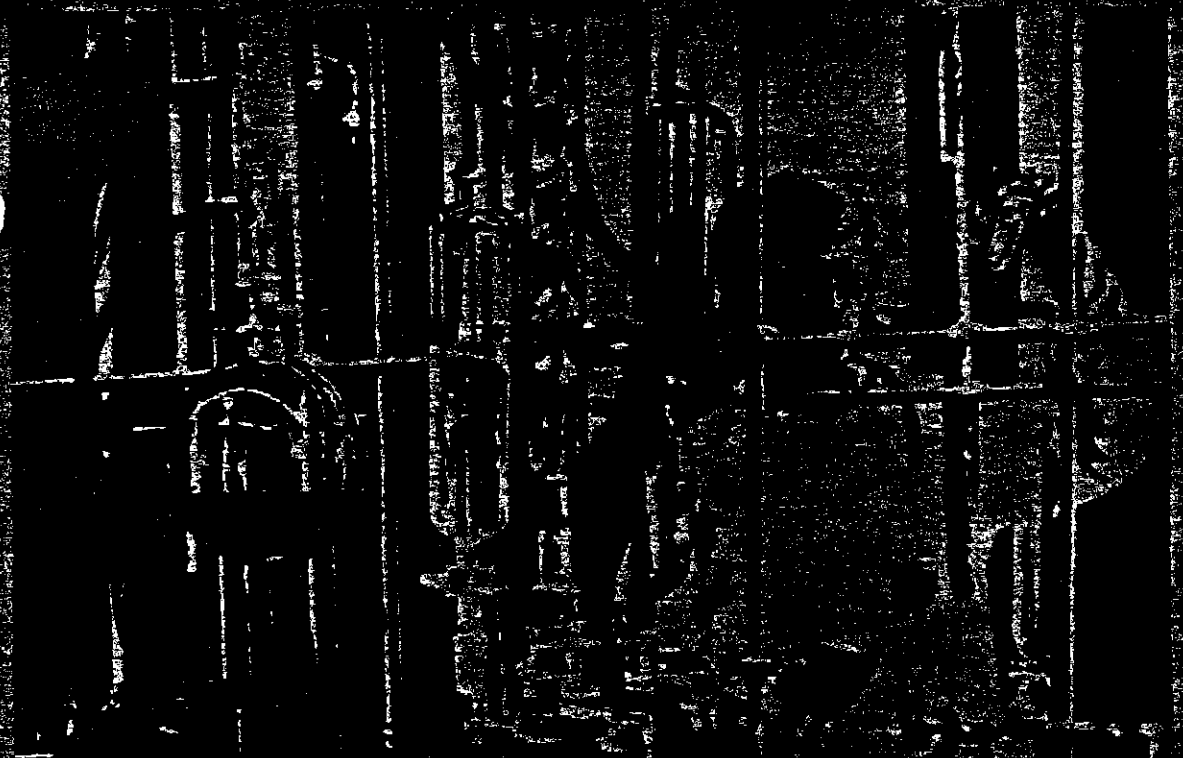
The location of the waste storage tank complies with the buffer zone requirements for underground tanks contained in the National Fire Protection Associations 1984 Flammable and Combustibles Liquids Code. The facility provides protection for exposures by location within the jurisdiction of the Washington Township Fire Department.

F-5f Management of Incompatible Wastes in Tanks

No incompatible wastes are managed in tanks at this facility.

Research & Development

Safety Manual



EMERGENCY PHONE NUMBERS

Fire, Accident, Medical, Escaping Gas 3113

Security, Theft 3103

Poison Control Center (Columbus) ... 9 228-1323

Ashland Chemical Company

Division of Ashland Oil, Inc.

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OUR CORPORATE SAFETY POLICY

Ashland's corporate safety policy is aimed at zero accidents. The goal of Venture R&D and of Operating Division's R&D is to improve our present record and to move towards that zero accident target. We are committed to the belief that Ashland employees should have safe jobs and safe places in which to work. The company will do its part to achieve a safe working environment by conforming to established safety standards within the guidelines of federal, state, and local codes and regulations. The company will continue to:

Design safety into all construction, new equipment, and processes.

Purchase safe and approved equipment and tools.

Furnish appropriate protective clothing and equipment.

Provide training and instruction in safe operations and work practices and encourage training of new employees by the more experienced employees.

Establish and enforce rules and regulations to promote safety.

As an important member of this operation, you have the obligation and responsibility for your own safety as well as that of your fellow employees. You must act in accordance with good, safe practices and procedures. Ashland expects all employees to conduct themselves in a safe manner at all times and to abide by the existing regulations, including the use of protective equipment. Under federal regulations, the company must enforce all safety rules and procedures.

Please report all injuries, accidents, and unsafe conditions or acts immediately to the appropriate supervisor. Give your full attention to safety. With your cooperation, we can eliminate the great personal loss that is involved in accidents.

Signed _____

Vice President
Venture Research

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SAFETY INFORMATION

FOR

LABORATORY AND NON-LABORATORY

PERSONNEL

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SAFETY IN GENERAL – YOURS IN PARTICULAR

This book is about staying safe. It is based on our belief that there is a safe way to do all jobs. The keys to accident prevention in the laboratory are understanding, foresight, and care, not just on the part of the laboratory workers, but by all personnel whose daily activities bring them in contact with the laboratory environment.

Each employee has a responsibility to self and co-workers to be fully aware of the safety requirements in the R&D area. Each should remain alert for special warnings or notices that may be posted for unusual situations.

The following rules are general and apply to everyone in the R&D area, visitors and employees alike:

> Eye protection must be worn in all laboratory and pilot plant work areas. Contact lenses are not allowed as they tend to trap harmful material in the eyes; any exception to this rule must be approved by the Secretary of the Safety Committee.
>
> Smoking is not permitted in pilot plants or laboratory work areas.
>
> Storage, preparation, or eating of food is prohibited in the laboratory. Do not use laboratory glassware for handling or containing food, and store food only in refrigerators specifically marked "FOOD ONLY".
>
> Laboratory benches, fume hoods, aisles and storage areas must be kept clean and free of clutter. Chemical spills must be cleaned up immediately.
>
> Safe laboratory practices are mandatory: chemicals are to be handled and disposed of properly, adequate safety equipment employed, operating equipment appropriately tagged, etc.
>
> Sandals or open-toed shoes may not be worn in the laboratory or pilot plant areas.
>
> And, of course, horseplay is strictly forbidden.

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Expanding a little on the preceding rules, smoking and eating are permitted in designated areas only. These rules apply uniformly to all laboratories regardless of the work being performed. The permissable areas include laboratory offices unless flammable or toxic vapors are present. Be especially alert for visitors to the laboratories and be sure they are equipped with safety glasses (available from the receptionist or guard) and are not carrying smoking materials.

If you are a laboratory worker, you should fully understand the properties of materials you are working with and the procedures you are using. For information regarding a specific chemical, refer to the appropriate Material Safety Data Sheet, available from your supervisor. Anticipate and avoid potential hazards. Know what dangers are involved in your activities, and inform others in your work area. Equally important, familiarize yourself with the location and use of emergency facilities and equipment; for example, fire pull station, fire hose, fire extinguishers, fire blankets, chemical showers, eye wash, first aid kits, etc.

SAFETY EQUIPMENT

Suitable eye protection in the laboratory usually means safety glasses, but can also include goggles or a face shield, depending on the situation. As stated elsewhere, all visitors as well as employees are required to wear eye protection. Safety glasses for visitors are available from the R&D receptionist or guard.

The company will provide prescription or plain safety glasses to all employees who work in laboratory or pilot plant areas. The secretary of the Research Services Coordinator will order your glasses and arrange for them to be fitted by an optician.

Until you have safety glasses, wear goggles, available in the stockroom, over your regular glasses. Plastic side shields are available for added protection against splashes. Contact lenses may not be worn in the laboratory, as they tend to trap harmful materials and vapors in the eyes. Any exception to this rule must be approved by the Secretary of the Safety Committee.

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Pilot plant personnel and many laboratory personnel need special safety shoes to protect from spills and dropped objects. You may order safety shoes through the secretary of the Research Services Coordinator. Upon written approval by a research manager, the company will contribute up to $30.00 toward the cost of safety shoes; if the selected shoes cost more than $30.00, you will be required to sign a payroll deduction authorization for the difference. An employee who has been issued safety shoes must wear them at all times in the laboratory or pilot plant.

Laboratory coats to protect your clothing may be ordered through the stockroom clerk. These coats are NOT fireproof and provide only limited protection from spills. Uniforms and bath towels are issued by the stockroom clerk to pilot plant personnel. Disposable paper towels are available from the stockroom.

Gloves, special protective clothing, safety shields, and other personal protective equipment are available as required from the stockroom. Make sure you know the proper type.

For emergencies, fire extinguishers and safety showers are located in all laboratory areas. Fire hoses, fire blankets, eye wash fountains and first aid kits are strategically placed throughout the R&D Building.

In a fire or oxygen deficient atmosphere, self-contained breathing apparatus (Scott Air Paks) must be used. Self-contained breathing apparatus are to be used only by employees who have been specifically trained in their use. Whenever a Scott Air Pak is used, notify the engineer at Extension 3110.

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EMERGENCIES

Two types of emergencies are considered here:

- Injuries and Illnesses
- Fire, Fumes Requiring Building Evacuation

A. Injuries and Illnesses

In case of minor injury or illness, alert your supervisor or a colleague, go the the first aid room located on the first floor near the northwest lobby, alert the Research Services Coordinator or the Research Manager - Staff; these, in turn, will alert members of the rescue squad trained to render first aid. If needed, a decision will be made to send you to the doctor or the hospital.

In case of serious injury or illness, do as follows:

1. Call 3113, explain the nature and location of the emergency and ask for the ambulance.
2. Contact your Supervisor and the Research Services Coordinator or the Research Manager - Staff.

In case of doubt, assume that an injury or illness is serious.

Following are some general guidelines which should be useful in case of injury or illness:

- Do not move an injured person unless there is a danger of loss of life. A stretcher is available in the First Aid Room for emergency use.
- In case of burning clothing or chemical spill on body, deluge the injured body area immediately under a safety shower, removing any clothing contaminated with chemicals. Seconds count, so don't waste time because of modesty. Keep the water running on the injured area for a least 15 minutes to remove heat and to wash off chemicals. Resume if pain returns. Wash off chemicals with a mild

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detergent and water but do not use neutralizing chemicals, unguent creams, lotions, or salves. Place clean, soaking wet, ice-packed cloths on burned area, and wrap the victim to avoid shock and exposure. Get medical attention promptly by calling Extension 3113.

Do not use a fire extinguisher on a person whose clothing is burning; he may suffocate. Do not use a fire blanket to smother the fire if a safety shower is nearby. The blanket does not cool, and smoldering clothes continue to burn. Any chemicals on skin or clothing will still be present and active. However, the blanket may be used to assist the injured en route to and after the use of the shower, and it is certainly better than nothing.

- In case of sudden illness, if the victim is a laboratory worker, or has been around chemicals, some form of poisoning should immediately be suspected. Be certain to find out exactly what substances may have been inhaled or ingested. If a toxic gas is involved, move the victim quickly to fresh air. A victim who has swallowed a chemical should be given a glass of water immediately, and Extension 3113 should be called for medical assistance.
- In case of chemicals in the eyes, immediately flush the eye with copious amounts of water under gentle pressure from an eye-wash fountain or spray. Check and remove contact lenses at once (as mentioned, use of contact lenses is not allowed in the laboratory unless approved by the Secretary of the Safety Committee). Hold the eye open to wash thoroughly behind the eyelids. In the absence of eye-wash fountain, place the injured on his back and gently pour water in the eye. The injured eye must be held open, and the victim given prompt medical attention, regardless of the severity of the injury. Keep the eyes immobilized with clean, wet, soft, cold pads while transporting the injured to medical personnel.

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- In case of limited contact of chemicals on the skin, immediately flush the contaminated skin area with cold water for 15 minutes and wash with a mild detergent, preferably soap, and water. If there is no visible burn, scrub with warm water and soap, removing jewelry to facilitate removal of residual chemical. If any damage is suspected, get medical attention. If a delayed reaction is noted (sometimes as late as the next day), report immediately for medical attention and explain carefully what chemicals were involved.
- In case of cuts, if blood is spurting, place a pad directly on the cut, apply firm pressure, wrap the victim to avoid shock, and get immediate medical attention. In the case of a less severe cut, wash, remove any pieces of glass, wrap the injured party to avoid shock, except in the case of trivial cuts, and get medical attention. A pressure pad may be applied firmly on the wound. NEVER USE A TOURNIQUET. If the cut is minor, wash it at the sink and scrub off any chemicals with soap and warm water, then get medical attention regardless of how trivial the wound seems.

In case of any type of accident or injury, members of the Rescue Squad are trained to render first aid while awaiting professional care.

B. Fire, Fumes Requiring Building Evacuation

If a fire, gas leaks, or unusual odors get out of control and there is a need for building evacuation, do as follows:

1. Pull alarm (this will automatically summon the Dublin Fire Department).
2. Call 3113, explain nature and location of emergency.
3. Evacuate the building.
4. Assemble in front of building.
5. Report to your supervisor.

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The general EMERGENCY PROCEDURES which have been distributed to all employees must be followed; extra copies are available from the Secretary of the Safety Committee.

The following guidelines should be useful for handling such emergencies:

- Supervisors will provide instruction to all of their people on how to use the various types of fire extinguishers, and any employee who has not received such instruction should arrange to get it. Notify your supervisor and attend instructions when they are available; these are set up by the Research Services Coordinator.
- Look at the fire extinguishers in your area to familiarize yourself with their location, type, and operation. All fire extinguishers are sealed; it is necessary to pull a pin and break a seal before an extinguisher can be used. Most are operated by squeezing the handle or a trigger and aiming the nozzle at the base of the flames.
- Small fires, when handled quickly, can usually be extinguished with fire extinguishers by personnel in the immediate vicinity. Normally, you can approach a small fire to within 3 to 6 feet, depending on the type of extinguisher. But the closer the approach, the greater the hazard. Always keep close to an exit in case flames spread faster than expected, and don't allow the fire to come between you and the exit. Never turn your back on a fire, even if it appears to be extinguished. No one is expected to fight a fire at great personal risk. If the fire gets out of control, follow the emergency procedures. Immediately after the fire, call Extension 3110 and ask to have used extinguishers refilled. Never return a partially full extinguisher to its rack.
- All fires, no matter how small, must be reported to the Research Services Coordinator or to the Research Manager - Staff.

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- Gas escaping from the pipelines throughout the building is a danger, both through toxicity and the possibility of explosion and fire. Detection of the odor of gas should start an immediate search for the source, which may often be an open valve. If the leak cannot be found quickly, follow the emergency procedures.
- If you detect an unusual odor, report it to your supervisor. You and your supervisor should then attempt to determine if the odor is originating in your area. If the odor source cannot be located, your supervisor should report the odor to the Research Services Coordinator or the Research Manager - Staff. After the problem has been identified, you will be informed of the cause of the odor and any steps which have been taken to assure that the problem does not reoccur. If the problem gets out of control, follow the emergency procedures.

BUILDING EVACUATION

When you hear the fire alarm, you must do the following:

- Rapidly, but calmly, secure whatever you are working with so it is safe to leave.
- Take fire extinguishers from your room into the corridor, laying them down against the wall, so they will be readily available to fire fighters.
- Set fume hood controls at "slow" unless material in the hood necessitates full draft for safety reasons.
- Turn off running water not needed for safety.
- Close all doors, and leave the building by the nearest exit taking you away from the fire. Use stairways. Do not use elevators.
- Assemble in front of the R&D Building.
- Report to your supervisor.

As soon as the engineer is advised of the location of the fire, he will relay the information over the loudspeaker system. Announcements can be heard in most

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parts of the building. However, these announcements are primarily for the members of the Rescue Squad. Others should not go to the scene of the emergency, but rather should try to avoid the area when leaving the building. Only Rescue Squad members should deliberately go near the area. When evacuating the building, if the location of the fire is not known, it is best to avoid exits closest to the Solvent Storage Room or the Chemical Pilot Plant, since these are the most hazardous areas where potential explosions would endanger people as they pass.

Because fire is a real and ever-present danger in a laboratory environment, periodic fire drills are required. Your conduct during a drill should be exactly as in a real emergency. If you have a guest when there is a fire alarm or fire drill, you should ask the guest to leave the building with you. If a meeting is in progress, the person presiding should call a short recess so all attendees can leave the building. In the case of a telephone conversation, you should inform the caller that a fire alarm has been sounded and that you must leave, but will call back later. (Incoming phone calls during a fire alarm will simply go unanswered.) Remain outside the building until the "all clear" has been sounded. After a fire drill, or a genuine emergency which proved to be relatively minor, be sure all unused fire extinguishers are returned to their proper racks. Remember to have partially used fire extinguishers refilled immediately.

HIGH WINDS OR TORNADOES

If we have exceedingly high winds or a tornado in the area, an announcement will be made over the loudspeaker system. Follow instructions. Stay away from windows, and if evacuation becomes necessary, go quickly and calmly to the interior stairways of the building, avoiding

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exterior glass walls, and proceed to the basement. Try to find your supervisor, and wait until an announcement is made for an all-clear signal.

BOMB THREATS

If you receive a bomb threat by telephone, try to obtain answers to the questions below, listen for indications of the source of the call, then call the Security Department on 3103 immediately. If there is no answer, call the Security Department in Ashland, Kentucky, on 7–5701. Complete Form 7672, "Bomb Threat Call Checklist", and submit it to the Security Department as soon as possible.

Record the exact language of the threat and descriptive information on the caller (male, female, adult, child). Make a note of any speech patterns (slow, rapid, broken, sincere, normal, accent, excited, intoxicated, loud, familiar, disguised, impediment). Record any background noises (music, talking, traffic, airplanes, machinery, laughter, bar room, typing, being coached). Ask the following questions:

- WHEN will the bomb go off?
- WHERE is it located?
- WHAT type of bomb is it?
- WHAT does the bomb look like?
- WHY are you doing this?
- WHO are you?

A copy of Form 7672 is attached.

The proper handling of a bomb threat is the responsibility of the Security Department. You will be notified in due time if there is a need for the evacuation of the building.

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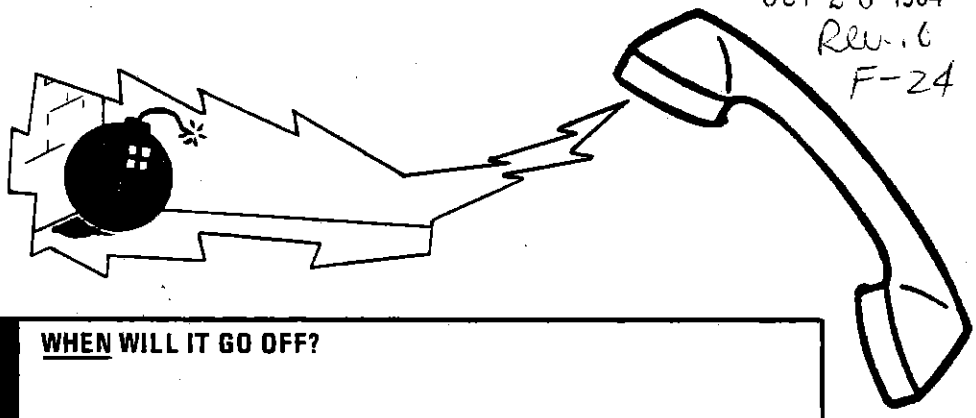
'OMB THREAT CALL CHECKLIST

CORPORATE SECURITY DEPARTMENT



WHEN	**WHEN WILL IT GO OFF?**
WHERE	**WHERE IS IT LOCATED?**
WHAT	**WHAT TYPE OF BOMB IS IT?**
WHAT	**WHAT DOES THE BOMB LOOK LIKE?**
WHY	**WHY ARE YOU DOING THIS?**
WHO	**WHO ARE YOU?**

SEE THE OTHER SIDE

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ACCIDENT REPORTS

In the event of any accident or fire, even if no injuries result, certain reports must be filed. The procedures are as follows:

- THE EMPLOYEE or someone who assisted with the emergency must report the accident immediately to the laboratory supervisor and to the Research Services Coordinator or the Research Manager - Staff.
- THE SUPERVISOR will investigate the accident and fill out Ashland's Form 136 and OSHA Form 101, which are available from the Secretary of the Safety Committee. These forms should be filled out promptly and in detail, including a complete description of the accident, its cause, any injuries which resulted, and their treatment. These forms are completed and returned to the Secretary of the Safety Committee within two days.
- IF PROFESSIONAL MEDICAL ATTENTION IS OBTAINED for an employee as a result of an occupational injury or illness, an Ohio Worker's Compensation Form No. JTC-3 must be filled out and must be returned to the Secretary of the Safety Committee, along with the physician's bill, if available.
- THE SECRETARY OF THE SAFETY COMMITTEE keeps accident records for internal and OSHA files, notifies the Ashland Safety Department when accidents occur, and forwards Form 136 and the State Worker's Compensation form to Ashland's insurance agency for processing.

REMEMBER, EVERY ACCIDENT AND FIRE IS IMPORTANT AND MUST BE REPORTED WITHOUT DELAY.

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INFORMATION ESPECIALLY FOR LABORATORY PERSONNEL

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LABORATORY SAFETY

Safety practices for the laboratory are the most difficult to anticipate and put on paper, yet among the most important to overall safety of R&D personnel. Obviously, laboratory safety is a blend of attitude, knowledge, and common sense. No matter how much information we include in this section, it will be incomplete.

In the end, laboratory safety depends on you. Use good judgment. When you aren't certain of the hazards of a particular material, inquire through the Environmental and Occupational Safety Department or the Safety Committee. When unsure of the best or safest procedure for a particular operation, ask your co-workers or supervisor.

STORAGE AND TRANSPORT

Cylinders of compressed gases must be supported upright by appropriate straps or chains to prevent them from falling, and must be placed away from heat or open flame. Never roll a cylinder on its bottom or handle a cylinder by its valve, and always remember to replace cylinder caps when a cylinder is not connected, or is being moved. When moving cylinders, strap them to a properly designated wheeled cart. Cylinder carts are designed to insure stability and to prevent cylinders from becoming potential rockets through broken valve stems should they fall.

A supply of the most commonly used compressed gases is maintained by the receiving clerk (Ext. 3679). The receiving clerk also keeps a record of all cylinders of compressed gases in the building. Periodic checks are made to encourage the return of unneeded cylinders, whether full or empty, since daily demurrage is charged for most cylinders and often exceeds the value of the gas within a few weeks.

Heavy items should be stored as near the floor as possible. Avoid any storage which could invite a spill or fall, and never store toxic or corrosive materials above eye level. Apparatus and glass tubing should not project beyond the front shelf limits, and fragile equipment should be clamped down or otherwise protected from accidental breakage.

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Discard any container which is not clearly labeled as to contents. Do not store potentially reactive chemicals near each other, and keep only the minimum required quantity of flammable liquids and solvents on hand. Great care must be used when solvent-containing materials are put in refrigerators. Even at refrigerator temperatures, explosive concentrations may develop. All refrigerators in the laboratory must be of the explosion-proof or laboratory safe type.

Transport flammable liquid in an approved safety can fitted with a flash-arresting screen. Reagent-grade chemicals may be kept in glass containers, but never in quantities of more than one gallon, and stainless steel safety cans are preferred. Transport glass bottles in protective carriers that will fully contain the contents if the glass were to break. Always use grounding cables when transferring flammable material from one container to another. Wear protective clothing and safety glasses whenever flammable, toxic, and other hazardous chemicals are being handled.

Because of their unstable nature, peroxides and perchlorates require special handling. Minimum stocks for reasonably efficient operation should be kept in specially market refrigerators. Larger quantities are kept in the Peroxide Storage Building (Carl Coats is responsible for this building and should be consulted concerning storage there). Before using peroxides or perchlorates at all, be sure you are thoroughly familiar with their properties and with safe handling techniques.

Dangerous concentrations of peroxides are likely to occur if some materials, notably diethyl ether or tetrahydrofuran, are distilled, evaporated, or stored for long periods of time in the presence of oxygen. Partly full containers of ether should be discarded if the remainder will not be used within a few days; containers should be dated when opened. Most ether containers are stamped with a packaging date, and normal shelf life should not exceed six months from this date.

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CHEMICAL SPILLS

When a potentially hazardous chemical spill occurs, alert your lab neighbors and evacuate the area. Notify your supervisor. If the chemical is volatile and flammable and ignition occurs, attempt to extinguish the flames on your way out of the laboratory. However, do not linger in the danger area. If the fire is out of control, follow the Emergency Procedures.

Even if the chance of fire or exposure to toxic material is remote, contact your supervisor. You or a co-worker trained in the use of Scott Air Paks should clean up the spill using the following procedure:

1. Put on self-contained breathing apparatus (Scott Air Paks) and appropriate protecting equipment (gloves, boots, apron, etc.).
2. Neutralize the spill, if needed, using weak acids or bases as required.
3. Absorb with vermiculite (available from the Stockroom Clerk).
4. Place wastes in a large plastic bag or other appropriate container.
5. Contact the Research Services Coordinator for disposal instructions.

Spilled mercury should be immediately and painstakingly cleaned up, using a trapped vacuum line attached to a tapered glass tube similar to a medicine dropper. Cover small droplets in inaccessible spots with sodium polysulfide solution or powdered sulfur to convert the mercury to mercury sulfide. Care should be used to clean up all mercury, especially any near a source of heat. A mercury cleanup unit is available from the Research Services Coordinator or his secretary. Domestic vacuum cleaners must not be used, since they will only redisperse mercury through the air and increase the contamination.

You should always be aware of the properties of materials you are working with and should have ready access to reference materials if in doubt. Questions about proper procedures and safety precautions, whether for routine tasks or in a spill or other emergency, may be answered by calling the Environmental and Occupational Safety Department, Ext. 3138, or the Chairman of the Safety Committee.

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SAFETY OPERATING PROCEDURES

Compressed Gases

Even gases such as air or nitrogen are hazardous when compressed. Cylinders of gas must be treated with care. Never loosen cylinder valve fittings to ease turning the valve or for any other reason. It is important that you use the correct regulator fittings. Oxygen cylinders must never be fitted with regulators which have been used with other gases. Cylinder outlets are deliberately varied to minimize the chances of you carelessly attaching the wrong type of fitting. Use adapters only after the possibility of adverse consequences has been carefully considered.

If you use two step-down regulators in series on a cylinder, be sure that a proper relief valve is placed between the two for protection of the downstream regulator. Several types of reducing valves and needle valves are available in the Stockroom; examine these carefully before use to be sure they are in good condition and are suited to the task at hand.

When opening the cylinder valve, always stand to the side of the regulator and open the valve slowly.

Always close cylinder valves when not in use, even though a closed reducing valve or needle valve is attached. The pressure should be released from the reducing valve after closing the cylinder valve. Do not leave pressure on a gauge unless it is being used or the diaphragm may rupture.

Rubber or plastic hoses should be used with gas cylinders only if a suitable reducing valve assures that excessive pressures will not be encountered. Cylinders should never be left in service until the pressure is zero; they should always be considered empty and be replaced when they still contain a few pounds of pressure. Cylinders should be returned with the valves closed and protective caps in place.

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Handling Chemicals

Many chemicals are at least potentially harmful. Avoid direct contact. It is especially important that you keep chemicals from hands, face, and clothing, including shoes. Many substances are readily absorbed into your body through the skin, or by way of inhalation. Chemicals on your hands can also easily end up in your mouth or eyes. Keep your hands clean, and wash thoroughly with soap and warm water whenever accidental chemical contact occurs. If clothing is contaminated, remove immediately and clean or discard as appropriate.

When pipetting chemicals, do not pipette by mouth. Use a vacuum or a pipette bulb. In the same vein, laboratory chemicals should never be deliberately tasted or inhaled.

While most laboratory chemicals should be treated as potential hazards, a number of common materials are extremely toxic. These should be dispensed and handled only in an adequate hood that is working properly, using appropriate protective equipment. These substances include, but certainly are not limited to, the following:

carbon monoxide	hydrogen sulfide	dimethyl sulfate
chlorine	hydrogen telluride	arsine
hydrogen cyanide	mercury	phosphine
hydrogen selenide	phosgene	acrylonitrile
benzene	epichlorohydrin	

Keep hoods clean and uncluttered — an uncongested hood has demonstrably greater air flow. This will also help prevent accidents and will minimize cleanup in case you have a chemical spill.

When you are conducting a reaction which may result in a mild explosion, or when you work with a vacuum system (which may implode), use a safety shield. Either use a face shield that is sufficiently large and strong to protect your head and neck, or use a standing shield.

The standing shield is mandatory if a more vigorous explosion is possible. Shields which can be knocked down by an explosion need to be securely stabilized with weights or fasteners. Preferably, standing shields should be secured at the top. Eye protection must be worn even when using the shields.

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Radioactive materials used in the laboratory are hazardous materials that come under the authority of the Nuclear Regulatory Commission and must be used only in accord with its regulations. Dr. Marvin Deviney is the Radiation Safety Officer and should be consulted before any work with radioactive material is planned.

Assembly of Apparatus

Many accidents result from the improper placement or misuse of equipment. Use all apparatus properly. Before starting a reaction, be sure that the equipment is suitable. Use only equipment which is free of flaws, and inspect it before and during use. No equipment or apparatus should be used or tampered with until you have learned its proper use, limitations, and possible hazards.

Set up approved apparatus, firmly clamped and well back from the edge of your lab bench, with due regard to positions of other equipment, chemicals, and to other workers and their equipment. Choose apparatus sizes which can properly accommodate the experiment to be performed, allowing about 20% free space.

Apparatus attached to a ring stand should be positioned so that the system's center of gravity is over the ring stand base and not to one side, and as low as possible without hampering removal of burners, mantles, or baths. Keep mouths of test tubes, flasks, etc., pointed away from you and others. Use a shield when doing any vacuum or pressure work.

Stoppers should fit so that between 1/3 and 1/2 is inserted. Keeping your hands close together and protected with gloves or multi-folded cloth, gently but firmly twist the stopper in place. Avoid exerting any pressure on inserted glass tubes. Use ground glassware, if possible. Glass stoppers and joints should be clean, dry, and lightly lubricated, with the lubricant giving a streakless mirror connection. Never force a frozen glass fitting. Loosen by heating gently in a low flame or under hot water. If this fails, use the proper loosening device such as a stopcock puller. Protect your hands.

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All glass tubing and rods, including stirring rods, should be fire polished before use. Unpolished glass has a razor-like edge, which not only will lacerate the skin, but will cut into a stopper or rubber hose, making it difficult to insert the glass properly. After polishing or bending glass, allow ample time for it to cool, then touch it gingerly before grasping it to be certain it is not too warm. Mercury or alcohol type thermometers should not be used for stirring rods – the fragile bulbs are too easily broken.

Handle extremely cold or hot equipment with great care using gloves and tongs whenever possible. Employ safety shields when heating acids, alkalies, or other corrosive substances, or when working with high boiling liquids of any kind at high temperatures. A properly placed pan under a reaction vessel or container will confine spilled liquids in the event of glass breakage.

Overnight Operations, After-Hours Work

Chemical reactions must frequently be continued overnight, occasionally for extended periods. When this seems necessary, check with your supervisor for approval of the necessity and safety of the operation. Then, obtain a "Continuous Operation" sign from the Stockroom and mark it clearly with your name and telephone number in case there should be trouble. Guards are instructed to turn off any operating equipment without one of these signs. Each laboratory should also have the supervisor's name and telephone number affixed to the window on the lab door, so that security guards may notify the supervisor if there is a problem.

When the urgency of your project dictates overtime lab work, you must be especially aware of potential hazards. Technicians can work after hours only at the request of an immediate supervisor. Dangerous work should never be undertaken unless there is someone nearby capable of rendering assistance, if needed.

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WASTE DISPOSAL

The responsibility of each individual for the materials he or she discards must be emphasized, and your careful attention to disposal procedures will prevent hazardous conditions.

Disposal of laboratory waste can present immediate and long range hazards if appropriate caution is not exercised. The following outlines methods of disposal available in our R&D labs to assist you in acting responsibly and safely. In addition, other manuals such as Aldrich Chemical Company Catalog can be consulted for proper disposal methods.

Solid Trash

Trash cans and material marked as trash left in the hallway are emptied nightly by janitorial personnel. These materials are dumped into a compactor, and the compacted mass is then hauled away to a landfill as needed. Points to consider are:

- The janitorial service personnel have no chemical training.
- Bottles and cans alike are crushed in this process. Any contained flammable, reactive, or toxic materials will likely escape, and probably will leak from the compacted mass.
- Once trash leaves your hands it automatically goes through this process. Please make sure the materials you mark for disposal in this manner are safe for treatment as described.
- No liquid material is allowed in the compactor.

In case of doubt on how to handle waste solid chemicals, contact your supervisor or the Research Services Coordinator before doing anything.

Waste Organic Liquids

There are four kinds of waste organic liquids to be considered:

1. Halogenated liquids.
2. "Clean" waste solvents, i.e., waste solvents of low non-volatile content.

3. "Dirty" waste solvents, i.e., waste solvents of high non-volatile content such as resin and polymer solutions.

4. Reactive liquid waste chemicals such as vinyl monomers, isocyanates, etc.

Three different kinds of cans are available to handle the halogenated liquids, the "clean" liquid waste and the "dirty" liquid waste. The reason these liquid wastes have been differentiated is for us to be able to handle them in compliance with the EPA (Environmental Protection Agency) and RCRA (Resources Conservation and Recovery Act) rules and regulations.

When having to dispose of reactive liquid wastes, please consult your supervisor or the Research Services Coordinator for advice.

These cans are emptied on a regular basis or as needed by Ashland personnel.

Water Soluble Chemicals

Small amounts of non-toxic water soluble chemicals such as some inorganic salts, acetone, alcohol, etc., may be disposed of in the laboratory sinks; please make sure to dilute these chemicals with large quantities of water.

Toxic Materials, Peroxides, Questionable Cases

The disposal of highly toxic materials (such as dimethyl sulfate or cyanide), reactive materials, and questionable cases (such as solids or gels containing solvent) are the responsibility of the chemist in charge. He or she must personally supervise such disposal with the help of a supervisor or the Research Services Coordinator.

Some substances may be mixed with additional solvents and discarded in the appropriate waste solvent can; others must be neutralized by means of chemical reactions. Large quantities of hazardous waste, or questions about necessary procedures, must be brought to the attention of the Research Services Coordinator.

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PILOT PLANT SAFETY

The procedures and regulations applicable to the laboratories also apply to pilot plant operations; however, extra precautions are necessary to guard against additional hazard potential in the pilot plant.

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SAFETY MANAGEMENT

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SAFETY COMMITTEE

A Safety Committee meets regularly to consider all matters pertaining to safety, including the reports of safety inspectors and accident reports. Members of the committee conduct monthly inspections of the R&D laboratories and pilot plants. The chairman and vice-chairman of the committee are appointed by R&D Management to serve six-month terms, at which time the vice-chairman becomes chairman and a new vice-chairman is appointed. The committee has a permanent secretary. The members of the committee are appointed for annual terms, with one-half replaced each six months. Membership represents each area of the R&D Building.

Any matter pertaining to safety should be referred to a member of the Safety Committee so that it may be properly considered and appropriate action may be taken. Of course, an urgent matter requiring immediate attention should be taken up with those directly concerned. Minutes of Safety Committee meetings are sent to all supervisors, who are expected to circulate them to all persons under their supervision.

RESCUE SQUAD

Volunteer representatives from each area of the building are trained in first aid and rescue techniques to assist in the event of an emergency. Their primary function is to rescue, not to fight fires, although they may aid with extra fire extinguishers if the fire is small. Laboratory personnel should know who the squad members are in their area.

As a rescue squad member, you may be summoned individually for help in the case of minor injuries. Use good judgment, remembering that if your services are really needed, professional medical attention is also needed. Avoid calling an ambulance unnecessarily, but do not hesitate to call it (Ext. 3113) if the emergency appears to be serious. Keep your training up-to-date by attending review classes regularly.

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In the event of a major emergency, all Rescue Squad members will normally be alerted by the sounding of the fire alarm. An Information and Procedures bulletin has been developed by the rescue squad team in cooperation with the Dublin Fire Department. Remember that rescue squad activities are for trained rescue squad members only and should not be carried out by untrained people. If you would like to join the rescue squad, please contact the Research Services Coordinator.

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CONTINGENCY PLAN &

EMERGENCY PROCEDURES

ASHLAND CHEMICAL COMPANY

RESEARCH & DEVELOPMENT LABORATORY

EPA I.D. NUMBER

OHD042311209

Generator

Transporter

Storage

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CONTINGENCY PLAN AND EMERGENCY PROCEDURES

SCOPE & APPLICABILITY

The following contingency plan describes the facility's response to a fire, explosion, or release of hazardous waste.

The intent of the contingency plan is to satisfy the requirements of the U.S. EPA Resource Conservation & Recovery Act, the U.S. EPA Comprehensive Environmental Response, Compensation, and Liabilty Act (CERCLA), and the Federal Water Pollution Control Act.

This plan is kept on file at the plant site. The plan has been submitted to local police and fire departments, hospitals and state and local emergency response teams that may be called upon to provide emergency services.

This plan must be amended whenever:

(1) The facility permit is revised.

(2) The plan fails in an emergency.

(3) The facility changes in design, construction, operation, maintenance or in any way that increases the potential for fire, explosions or release of hazardous wastes or changes the response necessary in an emergency.

(4) The list of emergency coordinators changes.

(5) The list of emergency equipment changes.

This contingency plan must always be available for inspection by an EPA inspector.

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COORDINATION AGREEMENTS

The laboratory Contingency Plan has been distributed to the local and State emergency response teams who are listed below. The local police and fire departments have come to the lab and have reviewed and approved the plant emergency action plan.

A copy of the Contingency Plan has been sent to the following State and local agencies:

Dublin Police Department
P.O. Box 175
Dublin, OH 43017

Mr. Bob Graves
Vice President of Ambulatory Service
Riverside Methodist Hospital
3535 Olentangy River Rd.
Columbus, OH 43214

Ohio Environmental Protection Agency
Emergency Response Section
361 E. Broad St.
Columbus, OH 43215

Mr. Charles Layman
Asst. Director of Laboratory Administration
318 Ohio State University Hospital
410 W. 10th Ave.
Columbus, OH 44310

Bureau Chief, Gene Bostic
Washington Township Fire Department
6255 Shire-Rings Rd.
Dublin, OH 43017

Dr. Mark W. Stock
55 S. High St.
Dublin, OH 43017

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The Washington Township Fire Department is the primary emergency coordinator according to Ohio Law. The Fire Department will coordinate activities through the Dublin Police Department, local hospitals, and other agencies. Upon arriving at the site, the Fire Department will meet with the emergency coordinator from Ashland to review the hazardous waste records describing the waste stored at the lab at the time. Based upon DOT classifications, the Fire Department will determine whether evacuation down-wind of the release is required. The Fire Department will instruct the police on which areas should be evacuated, if necessary. The Fire Department will then determine how to properly fight a fire or spill. The Fire Department will have access to all of the fire fighting equipment located at the facility for their use.

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EMERGENCY COORDINATOR

The primary emergency coordinator of the laboratory is the Vice President, Venture F&D. However, in his absence both the Research Manager Services and Research Manager - Foundry Product & Chemical Systems will act as emergency coordinators.

In the absence of the above listed individuals, the individuals listed below are familiar with the Contingency Plan and the Emergency Procedures and are authorized to act as emergency coordinators.

In addition to local plant personnel, Ashland maintains a 24-hour emergency reporting operator in Ashland, Kentucky (1-606/324-1133). This operator has names and phone numbers of personnel in the Corporate Safety, Environmental, & Occupational Safety Departments who can be contacted in the event of an emergency.

The following is a current list of Emergency Coordinators in the order in which they will assume responsibility. In the event of a fire, explosion, spill or release of material the emergency coordinator and his alternates have the authority to commit the resources necessary to implement this contingency plan.

Title/Names & Address	Home Phone	Office Phone
Vice President, Venture Research & Development James D. Idol 89008 Park Ridge Ct. Worthingtoin, OH 43085	888-2230	889-3188
Research Manager - Services Michel E. Mullier 125 St. Julien Worthington, OH 43085	436-4768	889-3272
Research Manager - Foundry Products & Chemical Systems William R. Dunnavant 4507 Kipling Upper Arlington, OH 43220	451-8374	889-3279
Superintendent of Building Services Elmer H. Hanson 919 Kenmure Ct. Columbus, OH 43220	459-0652	889-3737
Research Services Coordinator Jerry W. Boone 3581 Paris Blvd. Westeville, OH 43081	891-4126	889-3487

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Title/Names & Address	Home Phone	Office Phone
Ashland 24-Hour Emergency Operator P.O. Box 391 Ashland, KY 41101		1/606-324-1133

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EMERGENCY PROCEDURES

Notification

In the event of an emergency situation plant personnel are instructed to notify the emergency coordinator. The emergency coordinator is responsible for insuring that all facility personnel, local authorities, Ashland Corporate personnel, and State and Federal authorities are notified. The individuals and agencies who must be contacted are identified in Table 1.

Identification Of Hazardous Wastes

The emergency coordinator will immediately determine the source and amount of a hazardous waste release. He will determine the extent to which the plant and surrounding area are affected by a release.

The identity of any material which is released is determined from the location and source of the release and from plant inventory records. The hazardous wastes which are in plant inventory are recorded on the waste manifests. Their hazardous characteristics are obtained from the waste profile sheets. If for some reason the released material cannot be identified a sample will be taken for chemical analysis.

Hazard Assessment

The emergency coordinator is responsible for assessing possible hazards to the human health and the environment. To assist him Ashland maintains a 24-hour emergency telephone operator who provides contact with company personnel in the Safety, Environmental, and Occupational Safety Departments. If the plant has had a release, fire, or explosion which could threaten human health or the environment outside of the facility the emergency coordinator will immediately notify the National Response Center (800/424-8802).

Plant Emergency Action Procedures

The plant emergency action plans for dealing with a fire, explosion, or a spill or release are described below.

1.0 General Response Procedures

These are the general procedures that personnel will follow for all emergencies.

1.1 The person discovering the emergency will alert all others in the area. Persons who are exposed to immediate risk of injury will be moved to safety.

1.2 Immediate action will be taken to end the emergency only if this can be done quickly and safely.

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1.3 The emergency coordinator will be notified of the emergency.

1.4 The emergency coordinator will request the following information.

What is the type of incident? Is it a fire, explosion, chemical spill or vapor release?

- What is the exact location and source of the emergency?

- What is the extent of the incident? How large is the area which is affected? How much material has been released or spilled? What is the material which has been released. He will request a sample if necessary.

What is needed in terms of equipment and people to combat the emergency?

1.5 The emergency coordinator will implement the plant contingency plan if necessary. This includes the following.

- Notify all plant personnel of the nature and location of the emergency. This includes confirming that the personnel who are responsible or needed for combatting the emergency have been committed.

- Decide if a general or partial evacuation is required and if so confirm that evacuation orders are given.

- Decide if assistance is required from the local police, fire, and emergency squads. If so, confirm that they have been notified.

- Notify Ashland Corporate Personnel of the nature of the problem.

- Determine if a release, fire, or explosion has occurred which could threaten human health or the environment outside of the plant. If so, immediately contact the National Response Center of the U.S. Coast Guard (800/424-8802) and State Emergency Response Authorities (800/282-9378). He will provide the following information.

  - His name and telephone number
  - Name & address of the facility
  - Time & type of incident (e.g. release, fire)
  - Name & quantity of materials involved, to the extent known
  - The extent of injuries, if any, and
  - The possible hazards to human health, or the environment, outside the facility

2.0 Fire And/Or Explosion

The hazardous waste storage area and underground waste storage tank are easily accessed by fire-fighting equipment and emergency vehicles. The R&D Rescue Squad members will assist in extinguishing small fires. These personnel will provide specific instructions to police, fire or emergency squads who may be responding to an emergency call.

The following actions will be taken in the areas affected by a fire or explosion:

1.0 The individual discovering the fire will sound the alarm and alert persons in the immediate area and Rescue Squad members.

2.0 If it is possible to put out the fire immediately he will do so.

3.0 He will immediately notify the emergency coordinator or the area supervisor who will notify the emergency coordinator.

4.0 The emergency coordinator will request the following information.

- The exact source and location of the fire.
- The size of the area affected by the fire.
- Are there any injuries to personnel?
- What is needed in terms of men and equipment to put out the fire.

5.0 The emergency coordinator will take the following actions.

- Confirm that plant fire fighting personnel are informed of the nature and location of the incident.
- Confirm that personnel are shutting down their operations.
- Give the evacuation order if necessary.
- Confirm that the local authorities who may be needed for assistance have been notified.
- Notify the Ashland Corporate Emergency Operator (606/324-1133) of the incident.
- If the fire or explosion threatens human health or the environment outside of the plant, the emergency coordinator will immediately notify the State Emergency (800/282-9378) Response authorities and the National Response Center (800/424-8802).

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All fire extinguishers in the buildings are clearly marked.

Scott Air Paks are to be used by trained individuals. Upon hearing the plant emergency alarm they are to gain access to and ready themselves with the Scott Air Paks. After the Air Paks are on and have been checked these men are to proceed to the emergency area and stand ready.

3.0 Spills Or Material Release

In the event of a chemical spill the individual discovering the spill will immediately report it to the supervisor. The supervisor will contact the Emergency Coordinator.

The Emergency Coordinator will request the source, location and identity of the spill. He will determine the quantity spilled, the rate of spill and the direction in which the spill is heading.

The Emergency Coordinator will inquire if any injuries have occurred and whether a fire or explosion has occurred or may occur.

The Emergency Coordinator will assess the magnitude and potential seriousness of the spill or release. If the spill lies within the company's emergency response capabilities the Emergency Coordinator will contact and deploy the necessary lab personnel. If the accident is beyond plant capabilities the Emergency Coordinator will contact the local police and fire departments, the Ashland Corporate Emergency Operator (606/324-1133), State Emergency Response (800/282-9378) authorities and the National Response Center (800/424-8802).

Those areas of the plant in which a potential spill may occur are identified in the Spill Prevention Control & Countermeasure Plan (SPCC) which is attached. The potential amounts of materials that may be spilled and the spill containment systems are described in Section 9.0 of the SPCC Plan.

In the event that a spill is not contained in a diked or curbed area the plant spill control plan will be implemented. The objective of the spill control plan is to isolate the spill, remove the released material, and clean up and decontaminate the area. This is described in Section 11.0 of the SPCC Plan.

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If a chemical spill results in the release of a toxic vapor, the individual discovering the spill will immediately notify personnel in the area and Rescue Squad members and sound the alarm. He will then notify the emergency coordinator of the location, source, and magnitude of the release.

The emergency coordinator will insure that evacuation orders are given for all plant personnel who are not responsible for combatting a toxic vapor release. They will be given instructions to evacuate upwind of the leak. All personnel know all exits in their departments and the alternatives that can be used if an exit is blocked.

Resque Squad personnel have been pre-assigned to combat a toxic vapor release. When notified these individuals will outfit themselves with Scott Air Paks and report to the scene of the emergency. The emergency coordinator will confirm that these personnel have been informed of the location, source and nature of the toxic vapor release.

The emergency coordinator will determine if surrounding areas may be affected. The National Weather Service can be contacted at 231-5212 for prevailing wind patterns in the area.

If surrounding areas may be affected the emergency coordinator will notify the local fire and police departments, State Response authorities, the National Response Center (800/424-8802) and the Ashland Corporate Emergency Operator (606/324-1133).

As called for by the Comprehensive Environmental Response Compensation and Liability Act of 1980 (CERCLA), Ashland will report a spill of one pound or more of any hazardous material for which a reportable quantity has not been established and which is listed under the Solid Waste Disposal Act, Clean Air Act, Clean Water Act or TSCA. The same practice will be followed for any substances which are not specifically listed in these acts but which can be classified as a hazardous waste under RCRA. For those substances for which a reportable quantity has been established a spill will be reported if the amount reaches or exceeds the reportable quantity.

Any hazardous substance spill will be reported if it reaches a storm sewer or navigable waters. If one thousand gallons of oil is spilled in a single event it will be reported.

The above spills will be reported to the State Environmental Emergency Operator (800/282-9378) and the National Response Center (800/424-8802).

4.0 Floods

The hazardous waste storage pad and underground waste solvent tank are not located within the 100 year flood plain. Therefore, no provisions are required for floods.

Prevention Of Recurrence Or Spread Of Fires, Explosions, Or Releases

The R&D Safety Manual specifies that all personnel are to secure their operations after the plant emergency alarm is sounded. The emergency coordinator will monitor all equipment for leaks, pressure buildup, gas generation or ruptures.

All aboveground storage tanks are in curbed, diked areas. All spills in these areas will be contained until they can be collected and removed. Any spills outside of a containment area will be isolated and cleaned up. The collected material will be disposed at a permitted disposal site.

Any evidence of a leak in the underground waste solvent tank will require further study to ascertain whether this is an actual leak or an error in reading the gauge.

The contents of all damaged containers will be placed into empty drums.

Storage & Treatment Of Released Material

Immediately after an emergency, the emergency coordinator will make arrangements for treatment, storage, or disposal of recovered waste, contaminated soil, surface water, or any other contaminated material.

Incompatible Wastes

The emergency coordinator will ensure that no wastes are stored in an area that may be incompatible with a released material until cleanup procedures are completed.

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TABLE ONE

EMERGENCY CONTACTS

Emergency	Organization/Agency	Emergency No.
Fire/explosion	Fire Department	889-8382
	Police Department	889-1112
	Ashland Emergency Operator	606/324-1133
In case of a major fire/explosion which threatens surrounding areas	State Emergency Response Operator	800/282-9378
	National Response Center	800/424-8802
Hazardous material spill/release	Fire Department	889-8382
	Police Department	889-1112
	Ashland Emergency Operator	606/324-1133
All spills or releases which pose an immediate threat to human health or the environment outside of the plant.	State Environmental Emergency Response Operator	800/282-9378
	National Response Center	800/424-8802
Potential flood	U.S. Army Corps Of Engineers	513/684-3001
	National Weather Service	231-5212
Injuries	Fire Department	889-8382
	Hospital	261-5501

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SPILL PREVENTION CONTROL & COUNTERMEASURE PLAN

Onshore Research & Development Facility

Address: Ashland Chemical Company

Owner/Operator: Ashland Oil, Inc.
1401 Winchester Avenue
Ashland, Kentucky 41101

Contact Person: Jerry W. Boone

Management Certification: This SPCC Plan will be implemented as herein described:

Signed: _____
Michel E. Mullier, Research Manager

P.E. Certification: I hereby certify that I have examined the facility plan and being familiar with the provisions of 40 CFR Part 112 attest that this SPCC Plan has been prepared in accordance with good engineering practices.

Signed: Eldon E. Ronning

Print Name: ELDON E. RONNING

Registration No.: E-49075 State: Ohio

Date: 10/26/84

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SPILL PREVENTION CONTROL & COUNTERMEASURE PLAN

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1.0 APPLICABILITY

This SPCC Plan describes this facility's preparedness to prevent the discharge of oil and release of product or hazardous waste to the environment. In the event of a release the SPCC Plan describes the facility's response to control and remove the released material.

The intent of the SPCC Plan is to satisfy the requirements of the Federal Water Pollution Control Act [Section 311 (j) (1) (c)] the U.S. EPA Resource Conservation & Recovery Act (RCRA) and the U.S. EPA Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

This SPCC Plan was prepared to satisfy EPA regulations on Oil Pollution Prevention (40 CFR Part 112) and on Contingency Plan & Emergency Procedures for Hazardous Waste Facilities [40 CFR Parts 264.52 (a) & 265.52 (a)].

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2.0 REQUIREMENT FOR PREPARATION/IMPLEMENTATION
(Section 112.3)

Owners or operators of onshore and offshore facilities in operation on or before March 26, 1976 that have discharged or, due to their location could reasonably be expected to discharge oil in harmful quantities as defined in 40 CFR Part 110, into or upon navigable waters of the United States or adjoining shorelines, shall prepare a Spill Prevention Control and Countermeasure Plan (SPCC Plan) in accordance with 40 CFR Part 112.7.

No SPCC Plan shall be effective to satisfy the requirements of this part unless it has been reviewed by a Registered Professional Engineer and certified by such Professional Engineer. By means of this certification the engineer having examined the facility and being familiar with the provisions of this part, shall attest that the SPCC plan has been prepared in accordance with good engineering practices.

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3.0 AVAILABILITY OF THE PLAN (Sec. 112.4)

The SPCC Plan must be maintained on file at the plant. The plan must always be available for on-site review by employees of the EPA/state during normal working hours.

This plan along with any amendments will be submitted to the Regional Administrator of the U.S. EPA and to the appropriate State agencies in charge of pollution control upon their request following a reportable release of a hazardous substance from this facility.

This plan along with any amendments must be submitted to the above mentioned agencies whether requested or not within 60 days after either of the following oil spill events:

(1) A discharge of more than 1000 U.S. gallons of oil into navigable waters in a single spill event; or

(2) A discharge of oil in harmful quantities, as defined in 40 CFR 110, into navigable waters in two reportable spill events within any 12 month period. (40 CFR 110 defines a harmful quantity of oil as that which violates applicable water quality standards or causes a film or sheen upon the surface of the water or adjoining shoreline.)

Within 60 days of the occurrence of either of these two conditions, we must submit to the EPA Regional Administrator the following:

(1) Name of the facility;

(2) Names of the facility owner/operator;

(3) Location of the facility;

(4) Date of initial facility operation;

(5) Maximum storage or handling capacity of the facility and current normal daily throughput;

(6) Description of the facility including maps, flow diagrams, and topographical maps;

(7) A complete copy of the SPCC Plan with any amendments;

(8) The cause of such spill including a failure analysis of the system in which the spill occurred;

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(9) The corrective actions and/or countermeasures taken, including an adequate description of equipment repairs and/or replacements;

(10) Additional preventive measures taken or contemplated to minimize the possibility of recurrence;

(11) Such other information as the Regional Administrator may reasonably require pertinent to the plan or spill event.

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4.0 AMENDMENTS TO THE PLAN (Sec. 112.5)

This plan will be amended for any of the following reasons:

(1) When required by the EPA after review of the SPCC Plan which has been submitted because of either of the oil spill events described in the previous section.

Note: The EPA must submit a written notice of proposed amendments. Within 30 days from receipt of notice, we can submit written information, views and arguments on the proposed amendments. After considering all relevant material presented, the Regional Administrator will notify us of any amendments required or shall rescind the notice. We can appeal the decision of the Regional Administrator. The appeal must be made in writing within 30 days of receipt of notice.

(2) Whenever there is a change in facility design, construction, operations, or maintenance which materially affects the potential for a spill.

(3) The owner or operator is required by law to review each SPCC plan at least once every three years, and an amendment is required if such review indicates more effective control and prevention technology will significantly reduce the likelihood of a spill event (and if such technology has been field proven).

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5.0 CIVIL PENALTIES (Sec. 112.6)

Owners or operators of facilities subject to Section 112.3 (a) who violate the requirements of this Part 112 by failing or refusing to comply with any of the provisions of Sec's. 112.3, 112.4 and 112.5 shall be liable for a civil penalty of not more than $5,000 for each day such violation continues.

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6.0 FACILITY DESCRIPTION

Refer to the attached plot plan for the plant layout.

This is a Research and Develoopment Laboratory. Chemicals and solvents are shipped to the laboratory primarily in drum quantities by truck. These chemicals are stored in drums in the drum storage area of the lab.

Finished products made in the lab are also stored in holding tanks which are listed on the tank list attached to the Spill Plan. The underground waste solvents tank collects wash solvents from the pilot plant cleanup of reactors. This mixture is sold for beneficial reuse.

All waste solvents associated with lab operations are recovered. They are stored in drums or a solvent recovery tank. The waste solvents are either sold for beneficial use or shipped off-site to a solvent reclaimer or a waste disposal firm.

All waste drums are stored on the drum storage pad. Drummed wastes are stored and handled in the same manner as drummed products.

Spent wash solvents from the pilot plant and line flush solvents are stored in an underground solvent storage tank prior to their bulk shipment to an incinerator.

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7.0 SPILL HISTORY [Sec. 112.7 (a)]

The facility has experienced no reportable spill events within 12 months prior to the effective date of this part (circ. 1974) and therefore no written description or corrective action is necessary for preventing recurrence.

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8.0 SITE DRAINAGE

Refer to the plot plan for reference to plant drainage.

Surface drainage from the catch basins in the parking lot and the roofs of the buildings goes to Cosgray Ditch. This is normally limited to runoff from a heavy rainfall. Cosgray Ditch goes into the Scioto River east of the R&D lab.

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9.0 POTENTIAL SPILLS & CONTROL (Sec. 112.7)

The lab areas in which spills can occur are as follows:

1. R&D Lab dock
2. Drum storage pad
3. Tank farm
4. Pilot plant
5. Drum filling
6. Solvent storage room

The potential amounts of material that may be spilled in these areas and the means of containing the spill are as follows:

(1) R&D Lab Dock

Spills in the R&D lab dock could be caused from a leaking or punctured drum.

The maximum quantity of a spill would be one drum or 55 gallons for any one incident. A spill of this magnitude would be confined to the immediate area.

The dock floor is concrete and impervious. Absorbents would be used to pick up spills. Chemical neutralizer and absorbents will be used for acid and caustic spills.

(2) Drum Storage Pad

Spills on the drum storage pad could be caused from a leaking or punctured drum.

Once again the maximum quantity of a spill would be one drum or 55 gallons for any one incident. This spill would be confined to the storage pad.

The pad is constructed of 6" concrete and impervious. Absorbents would be used to pick up spills.

The north section of this pad is used to store waste drums. This area will be curbed and ramped to give the following containment for each of the sewer sections.

Bay #	Width	Length	Height	Gallons: Pad Vol	Ramp Vol	Pallet Volume	Drum Vol	Net Available Containment	Required Containment	% Extra Containment
1	12'9"	28'	9"	2003	197	194	264	1742	1654	5
2	12'6"	28'	9'	1964	193	194	264	1699	1630	4
3	12'6"	28'	9"	1964	193	194	264	1699	1630	4
4	12'6"	28'	9"	1964	193	194	264	1699	1630	4
5	8'6"	28'	9"	1335	131	97	132	1237	1029	20
6	8'6"	28'	9"	1335	131	97	132	1237	1029	20
7	9'7"	28'	9"	1508	148	97	132	1427	1134	26

Each section was designed to contain 10% of the contents of the drums plus rainfall for a 24-hr 100 year storm.

(3) Tank Farm

Spills in the tank farm could be caused from overfilling a tank or leaking of tank contents. Spills in the tank farm area are confined to the tank farm by the tank farm dike.

The largest tanks in the tank farm have a total volume of 3,000 gallons. If the entire contents of one of these tanks were spilled, the spill would be contained by the tank farm dike.

The tank farm dike has a capacity of 8,500 gallons which is well in excess of the contents of the largest single tank plus freeboard for precipitation.

Any spills in the tank farm will be cleaned up immediately. The recovered material will be shipped off-site either to a reclaimer or to a waste disposal firm.

(4) Pilot Plant

The reactors in the pilot plant are also a service of potential leaks due to vessel or pipe/hose failure.

The maximum amount of material that could be spilled would be 383 gallons, the size of the largest reactor. A spill of this size could be contained by diking and absorbents could be used for cleanup.

(5) Drumming Area

A spill could occur during drumming as a result of overfilling or due to equipment breakdown.

Operators are in constant attendance during drumming. They are available to immediately stop the operation if a spill occurs. The maximum spill which we would anticipate would be the contents of one drum (55 gallons).

There is a metal curb in the drum filling area that will contain the contents of one drum.

Any spills in this area will be cleaned up immediately. The recovered material will be shipped off-site either to a reclaimer or to a waste disposal firm.

(6) Solvent Storage Room

A spill could occur during transfer of the spent wash solvents to the underground waste solvent storage tank. The solvent storage room is diked on 4 sides with a ramp at the entrance door. There is no floor drain in the room.

Operators are in constant attendance during transfer and are thus available to immediately stop the pump if a spill occurs. The maximum spill anticipated would be the contents of one drum (55 gallons). The dike will hold 55 gallons.

Any spills in the solvent storage room will be cleaned up immediately. The recovered material will be shipped off-site either to a reclaimer or waste disposal firm.

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10.0 DESIGN & OPERATING INFORMATION

This facility conforms to the following design and operating standards.

(1) Facility Drainage

Drainage from the tank farm dike is controlled by a drain valve in the SE section of the dike wall. The drain valve is a manually operated open and closed type valve. The drain valve is kept in the closed position when not in use.

The dike is drained under responsible supervision. The accumulated material is examined visually before draining to be sure that no oil will be discharged. Analytical tests are made if necessary to ensure compliance with water quality standards. The drain valve is closed following drainage under responsible supervision.

(2) Bulk Storage Tanks

Bulk storage tanks which are used for storage must meet the engineering specifications of the Ashland Chemical Company. The engineering specifications are maintained by the Central Engineering Department which is located at Chemical Company headquarters in Columbus, Ohio. Engineering specifications are maintained for the bulk storage of flammable liquids.

No tank is used for storage of a chemical that does not meet the engineering specifications for that service.

Bulk storage tank installations are provided with dikes of sufficient capacity to contain the contents of the largest single tank plus freeboard for precipitation.

Drainage from the tank farm is controlled in the manner described in the previous section on facility drainage.

The solvent recovery tank is an 8000 gallon underground metal storage tank used to hold wash solvents from the pilot plant reactors.

All tanks are subjected to periodic integrity testing. This service is provided by the Plant Services group of the Central Engineering Department of Ashland Chemical Company.

The tank farm areas are inspected routinely. The tanks are inspected to detect corrosion or leaking of fittings and seams. Visible leaks which result in an accumulation in the diked area are promptly corrected.
The tank farm dikes and the areas around the dikes are also inspected routinely for signs of leakage such as erosion, wet spots or dead vegetation.

Since none of the storage tanks are equipped with internal heating coils, this is not a potential source of leaks.

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(3) Facility Transfer Operations

All piping must meet Ashland Chemical Company Engineering Specifications. The Engineering Specifications specify the material of construction and pressure and temperature ratings which are required for transfer piping.

Underground piping is buried in a 6-inch envelope of sand. Buried piping is protected from soil corrosion by a protective wrapping and coating. All materials and application are in accordance with the National Association Of Corrosion Engineers, Std. RP-01-69, Rev. '72.

The amount of cathodic protection needed is determined before installation either by calculation or previous experience or after the pipe is buried from measurements of pipe to soil corrosion potentials using a test rectifier. Ashland uses cathodic protection consultants for design of cathodic protection systems.

Pipeline terminal connections are properly identified and capped.

Pipe supports are designed in accordance with Ashland's Engineering Specifications.

Visual inspections are conducted regularly of aboveground valves and piping. Repairs are made as necessary.

Vehicular traffic would have to leave the roadway to damage any above ground piping.

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11.0 SPILL CONTROL PLAN

This facility has a comprehensive contingency plan of which the SPCC plan is only a part. The plant's contingency plan meets EPA regulations for Oil Spill Removal (40 CFR 109) and Contingency Plans for Hazardous Waste Facilities (Subpart D of 40 CFR Parts 264 & 265).

This part of the SPCC Plan describes the facility's response to a spill or release of product or hazardous waste. Refer to the plant's contingency plan for the responsibilities of the plant emergency coordinator and his alternates. The plant's contingency plan which includes the SPCC Plan has been sent to the state and local authorities who may be notified in the event of a spill or release.

In the event of a release or spill, this facility is prepared to commit all on-duty personnel to contain and clean up any spill at this or from this facility. The following "Spill Control Plan" procedure will be implemented.

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SPILL CONTROL PLAN

1. If possible, shut all valves immediately to cut off the source of the spill or release.

2. After this has been done or if this is not possible, immediately notify the area supervisor.

3. The area supervisor is to immediately notify the plant emergency coordinator or his on-the-scene alternate of the identity and amount of the spill or release.

4. The area supervisor is to inform the emergency coordinator of the personnel and equipment that he needs to contain a spill and clean it up.

5. All personnel on duty will report to the area supervisor and follow his instructions as to specific duties to be performed.

6. Move oil absorbent material, neutralizing material, and shovels from the R&D Lab area to the area of the spill.

7. Use shovels to move sand and dirt to encircle the spill and prevent it from entering a waterway, ditch, or sewer.

8. Use material to neutralize and/or absorb the spill.

9. Once a spill is contained, neutralized and/or absorbed, immediately remove it and clean the area.

   (A) Place all contaminated materials and recovered liquid in containers that are compatible with the material and that meet DOT requirements for shipping.

   (B) All hazardous waste or material contaminated with hazardous waste must be removed off-site for disposal at an approved disposal site.

10. Should a spill or release occur in other than normal working hours, the individual finding the spill will follow the emergency reporting procedure posted in the plant. If unable to reach the posted procedure in the plant, then telephone as follows:

    (A) Fire Department - 889-8382

    (B) Police Department - 889-1112

    (C) Ashland Oil, Ashland, Kentucky - 1-606/324-1133

    (D) Vice President, Venture R&D - 888-7436

11. Follow the attached reporting procedure for notifying the proper State and Federal authorities of a spill or release.

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REPORTING PROCEDURES FOR A SPILL OR RELEASE IN OHIO

National

Notification of a release shall be made as soon as practical once the necessary action is being taken to control the spill.

Duty Officer, National Response Center
U.S. Coast Guard
Washington, D.C.

800/424-8802

Ohio

Notify immediately by calling the 24-hour telephone number.

800/282-9378

Ashland

Report to Ashland Oil by the Emergency Reporting System

606/324-1133

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12.0 INSPECTIONS & RECORDS

EPA regulations for hazardous waste facilities (40 CFR Parts 264.15 & 265.15) and oil pollution prevention [40 CFR Part 112.7(e)(8)] require that a facility is periodically inspected. This facility has a written inspection procedure. This inspection procedure is described in the contingency plan.

Inspections are recorded in a log. The log is kept for three years from the date of inspection. Deteriorations or malfunctions revealed by the inspection are corrected before they lead to a spill or release of hazardous material.

The inspection logs include the following minimum information:

(1) The date of inspection
(2) The time of inspection
(3) The name of the inspector
(4) A notation of the observations made
(5) The date and nature of any repairs or other remedial actions.

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13.0 SECURITY

EPA regulations for hazardous waste facilities (40 CFR Parts 264.14 & 265.14) and oil pollution prevention [40 CFR Part 112.7(e)(9)] require plants handling hazardous wastes to be fully fenced. Entrance gates should be locked and/or guarded when the plant is not in production or is unattended.

This entire drum storage area is enclosed by a high security fence with locked gates. Entrance to the drum storage area is only through the gates and during non-attended periods the gates are locked.

All bulk storage tanks are located within diked areas. The dike drain valves are kept in the closed position.

Access to the underground waste solvent tank is the fill line which is located in the solvent storage room. The solvent storage room is locked during non-attended periods. The R&D Dock which is adjacent to the solvent storage room has television surveillance by the guard service.

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14.0 PERSONNEL, TRAINING, AND SPILL PREVENTION PROCEDURES

Ashland's Reserach & Development Laboratory has an active safety program which includes training required by the Resource Conservation & Recovery Act (40 CFR Parts 264.16 & 265.16) and by Oil Pollution Prevention Regulations [40 CFR Part 112.7 (e)(10)]. A written description of this facility's training program is contained in the plant contingency plan.

Training activities and personnel training records are maintained by the research manager. The research manager is the designated individual who is responsible for the Spill Prevention Control and Countermeasure (SPCC) Plan.

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15.0 TANK LIST

DUBLIN, OHIO R&D PILOT PLANT
TANK LIST

Tank Number	Tank Capacity	Product Stored
6	3	High Energy Fuel Hold
7	2	High Energy Fuel Hold
8	2	High Energy Fuel Finished Product
9	3	High Energy Fuel Blend
	8	Waste Solvents

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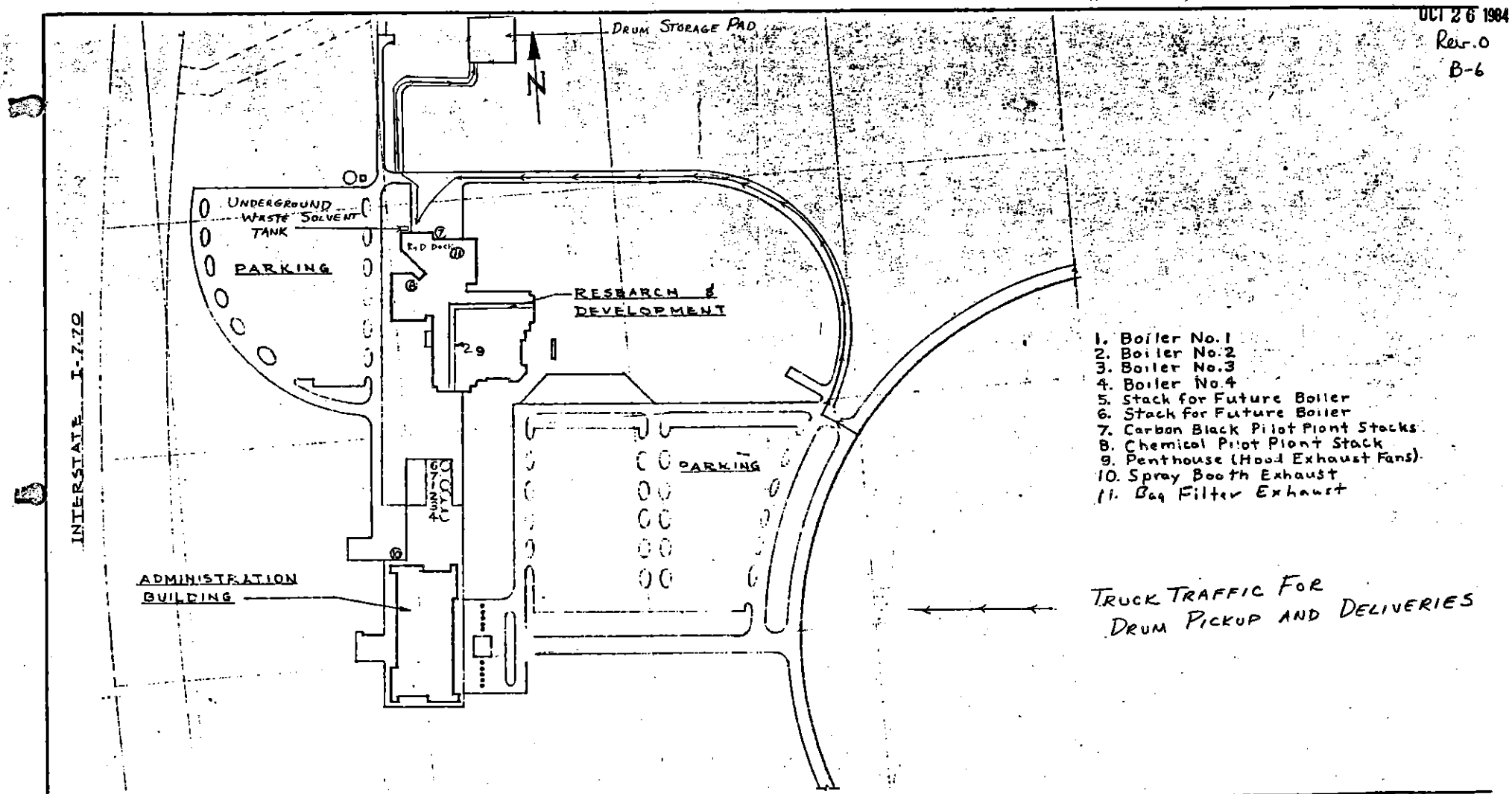

REV.	DATE	DESCRIPTION	APPR.

Ashland ASHLAND CHEMICAL CO.

Emission Sources
Plot Plan

SCALE | DR. BY
DATE | CK. BY

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EMERGENCY EQUIPMENT

A list of the emergency equipment which is available is attached.

Location: Dublin, Ohio

EMERGENCY EQUIPMENT

Physical Description	Location	Capabilities/Intended Use
Absorbant Materials	R&D Basement	Absorb spills
Air Compressor	R&D Basement	Transfer liquids
Air supply, for breathing equipment	2nd Floor R&D	Fill Scott Air Packs
Back Stretchers	2nd Floor R&D	Carry Injured
Bullhorn	1st Floor R&D Guard Station	Communications
Camera/Photo Equipment	3rd Floor Library	Take Photos of Emergency
Chain Hoist	1st Floor Administration Bldg.	Lift Equipment
Chemical Neutralizers	R&D Basement	Neutralize Spills
Explosimeters	1st Floor R&D	Measure explosive limits
Fire Extinguishers	Throughout the lab and pilot plants in well marked locations	Put out fire
First Aid Supplies	1st & 2nd Floor R&D	Treat Injuries
Foam Concentrate Supply	Chemical Pilot Plant	Put out fire
Foam Generators	Chemical Pilot Plant	Make foam
2 Forklifts	1- Chemical Pilot Plant 1-1st Floor Admin. Bldg.	Move Drums, Equipment, etc. Move Drums, Equipment, etc.
Food Supply	1st Floor Administration Building	Nourishment
Fuel Supply	Tanks between R&D and Administration Building	Provide Transportation
Geiger Counter	1st Floor R&D	Measure Radiation

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Physical Description	Location	Capabilities/Intended Use
Heaters, Portable	Basement - Admin. Building	Provide Heat
2 Inhalators	1-1st Floor R&D 1-1st Floor Admin. Building	Assist Breathing Assist Breathing
Jacks	1st Floor Admin. Bldg.	Lift Equipment
Lighting Equipment, Portable	Flashlight throughout R&D	Provide Light
Medical Supplies	1st Floor R&D 1st Floor Admin. Bldg.	Treat Injuries
Metal Saw (Power)	1st Floor R&D 1st Floor Admin. Bldg.	Cut Materials
Public Address System	R&D and Admin. Bldg.	Communications
Radio	1st Floor R&D 1st Floor Admin. Bldg.	Communications
Resuscitator	1st Floor Admin. Bldg. 1st Floor R&D	Aid in Breathing
Sand Supply	R&D Basement	
Sprinklers	Chemical Pilot Plant R&D	Extinguish Fires
Alarms	R&D and Admin. Bldg.	Communications
Personal Protective Clothing 2 Acid Master Suits 15 Scott Air Packs	1st, 2nd & 3rd Floors R&D	Protect Workers

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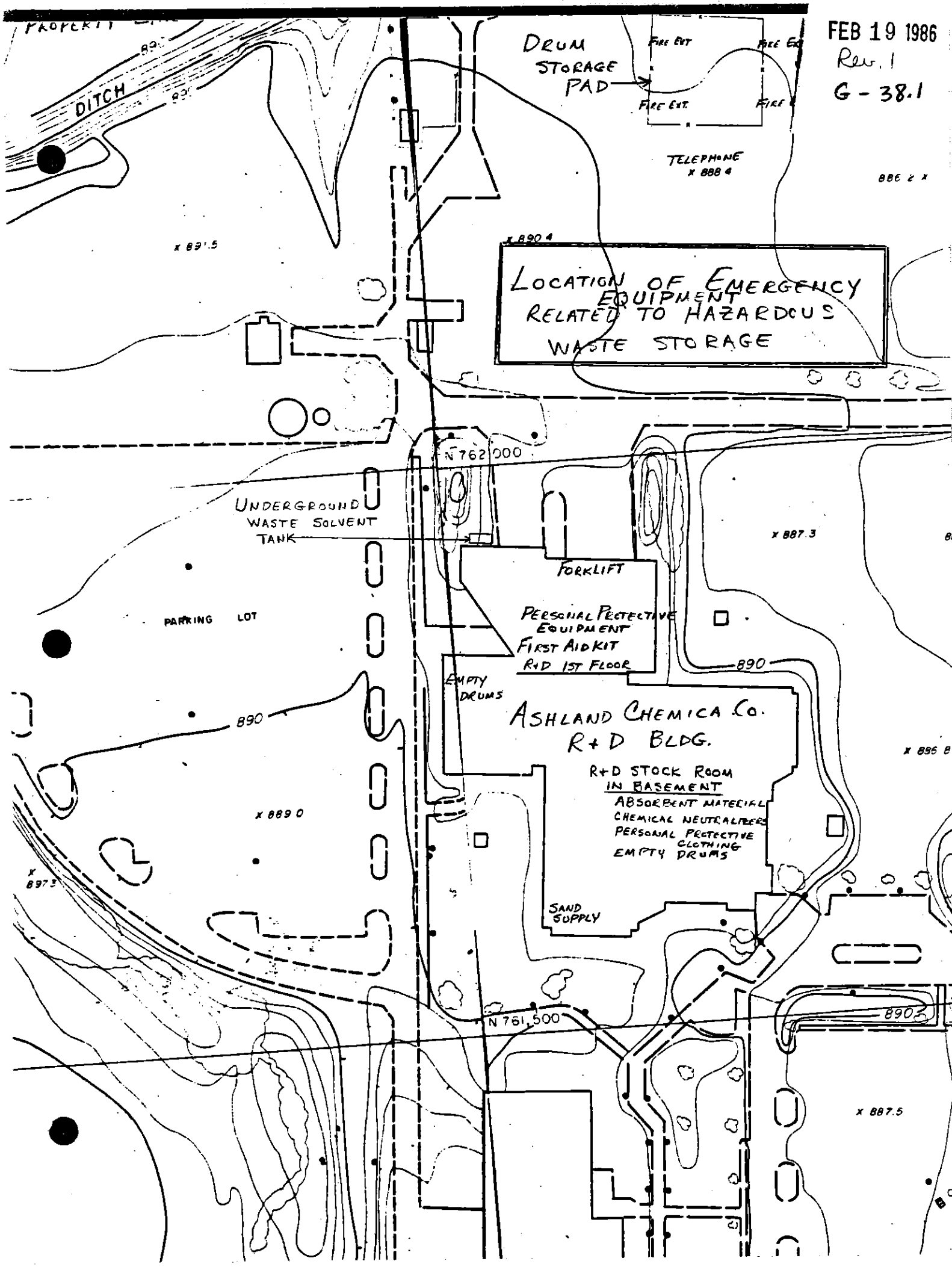

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ASHLAND CHEMICAL COMPANY

EVACUATION PLAN

I. In the event of a serious spill and/or fire or explosion, all personnel will immediately evacuate the premises.

A. The alarm will be sounded over the lab.

1. All employees, except rescue squad personnel, must evacuate the building by the nearest exit.

B. All exits are marked and all employees are familiar with the various routes of exit.

C. Upon complete evacuation of the plant, all employees will immediately assemble outside the front of the R&D building in the area shown on the map on the attached Emergency Procedures page.

1. The head of each group arriving at the assembly point will take attendance and report this information to the responding Fire and Police Departments. The authorities have received copies of this plan and are aware of this assembly point location.

2. All employees will stay at the assembly point until told otherwise by the authorities or the Ashland emergency coordinator.

II. Training

A. All employees will become familiar with the plant layout, fire exits and evacuation routes by conducting periodic fire drills and safety meetings.

1. Records of these drills and meetings are maintained at the plant.

B. In order for local authorities to become familiar with the plant layout, meetings and inspections are to be scheduled with local authorities as convenient.

1. Records of these meetings and inspections are to be kept at the plant.

III. General

A. The plant is so constructed that those areas that represent the greatest potential for a hazardous spill or fire are located away from the primary evacuation routes.

ASHLAND CHEMICAL RESEARCH & DEVELOPMENT

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EMERGENCY PROCEDURES

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For Serious Injuries & Illnesses

1. Call 3113 and explain the nature of the emergency.

2. Contact your supervisor and Mr. J. W. Boone (3487) or Dr. M. E. Mullier (3272).

For Fire, Fumes or Building Evacuation Emergency

1. The person pulling the fire alarm must call 3113 and give the operator the location of the emergency and the nature of the emergency. Tell the operator if there are any injuries.

2. All employees except rescue squad personnel must evacuate the building by the nearest exit and assemble outside the front of the R&D Building in the area shown on the attached map.

3. All Managers, Group Leaders and Supervisors must check with their personnel in the assembly area to be sure everyone is out the building. The front entrance security guard will stand near the assembly area with a bull horn to make any announcements.

4. Dr. Idol, Dr. Mullier (or Dr. Dunnavant in their absence) and Mr. Boone will pick up a walkie-talkie from the front entrance security guard. The rescue squad leader will pick up a walkie-talkie from the back entrance security guard. Security guards at the front and back entrances of the building will have master keys available for use by the Fire Chief.

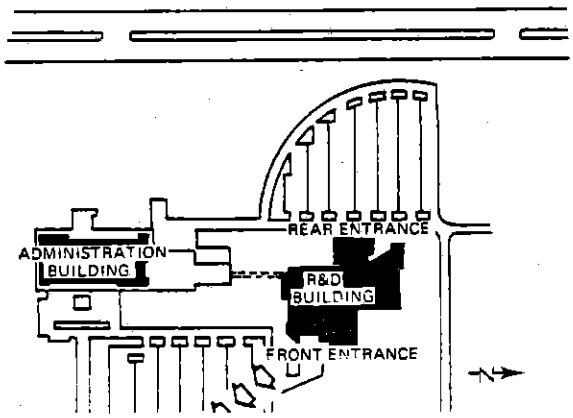
5. The rescue squad will assemble next to the first floor rear guard station. If this area cannot be reached because of hazards, the rescue squad will assemble at the first floor concourse junction.

6. Building Services will interrupt the fire alarm long enough to announce the location where the fire alarm was pulled and if 3113 was called, the nature and location of the emergency.

7. Dr. Idol, Dr. Mullier (or Dr. Dunnavant) and Mr. Boone will assemble and form a command post in front of the R&D Building.

8. After checking with the command post, the Rescue Squad will investigate the location and nature of the emergency and report by walkie-talkie back to the command post. After this has been completed, the Rescue Squad shall proceed without delay to systematically search and evacuate the building beginning at a point as close as possible to the location of the emergency. Cleared areas will be reported back to the command post.

9. Upon arrival of the Fire Chief, the command post will shift to the Fire Chief's location. The Fire Chief will take over operation of the command post.



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Ref: Sec. 264.73

RCRA OPERATING RECORD

A written operating record must be kept for all hazardous waste activities at the lab. These records must be maintained until closure of the part of the lab dealing with hazardous waste. The following information should be recorded when it becomes available:

(1) Description Of Each Hazardous Waste

A description of each hazardous waste received, stored and/or transferred by the facility. The description is logged in three ways: (A) common waste name, (B) the four digit U.S. EPA Hazardous Waste number, and (C) physical form of the waste.

(2) Dates

Date that each hazardous waste is stored at the lab, and/or transferred to a T/S/D facility or reclaimer.

(3) Quantity Of Each Hazardous Waste

The estimated or manifest-reported weight, or volume and density in one of the units of measure specified in the table below.

Units Of Measure	Symbol	Density
Pounds	P	
Short Tons (2,000 Lbs)	T	
Gallons (U.S.)	G	P/G
Cubic Yards	Y	T/Y
Kilograms	K	
Tons (1,000 Kg)	M	
Liters	L	K/L
Cubic Meters	C	M/C

(4) Manifest/Waste Profile Sheet

For each hazardous waste shipment stored and/or transferred the manifest number for the shipment is logged. In addition, for each waste the Chemical Waste Profile Sheet ID Mark is logged. The Chemical Waste Profile Sheet serves as a source of information used to classify the waste.

(5) Location Of Waste Storage

The location of hazardous waste stored in drum(s) is logged and shown on a plot plan. The method of storage is also identified in the log.

S01 - Container (barrel, drum, etc.)

The contents of the underground waste storage tank is also logged. The location of the tank is shown on a plot plan. The method of storage would be S02 - Tank.

(6) Records Of Waste Analysis

For each waste which a Chemical Waste Profile Sheet is completed. The chemist or chemical technician completes this sheet based upon knowledge of the contents of the drum.

A setaflash screening test is run on each drum in storage prior to manifesting and shipment. The purpose of these tests is to determine that the waste is consistent with that described on the chemical waste profile sheet.

If a significant discrepancy is found the individual generator is contacted immediately. If the discrepancy cannot be resolved with the chemist, further analyses will be run to characterize the waste.

(7) Summary reports and details of all incidents requiring implementation of the Contingency Plan. It must be noted in the operating record the time, date, and details of any incident that requires using the Contingency Plan. This should include the following information which must be submitted by written report to the US-EPA Regional Administrator and to the Ohio EPA within 15 days after the incident:

(1) Name, address, and telephone number of the owner or operator of the plant

(2) Name, address, and telephone number of the plant

(3) Date, time, and type of incident (e.g., fire, explosion, spill)

(4) Name and quantity of material(s) involved

(5) The extent of injuries, if any

(6) An assessment of actual or potential hazards to human health or the environment, if applicable

(7) Estimated quantity and disposition of recovered material that resulted from the incident

(8) Records and results of inspections. Inspection logs or summaries of all equipment related to hazardous waste management must be kept as part of the operating record.

(9) Closure cost estimates. All closure cost estimates required for treatment, storage or disposal plants will be kept as part of this operating record. A disposal facility must have a cost estimate for post-closure monitoring and maintenance.

(10) Manifest procedures. The specific manifesting procedures which will be used are as follows:

1. Ashland Research Services Coordinator completes the Uniform Hazardous Waste Manifest Form 8700-22(3-84) and the Continuation Form 8700-22A(3-84). For each waste, information must be provided as described in the Chemical Waste Management, Inc. Guidelines For Preparation of Hazardous Waste Manifests which is at the end of this section.

   The Research Services Coordinator makes certain that a chemical waste material profile sheet has been developed for each waste that is to be shipped. This insures that information is available to characterize each waste.

2. Before the driver removes waste he must count the number of drums or check the volume pumped into the tank truck to verify the total shown on the manifest. He must check the hazardous waste labels on the drums to see that they refer to the correct waste manifest number.

   When he is satisfied that everything is in order he signs and dates (Item 17) the manifest as the first transporter representative. He then provides Ashland with the generator copy and the remaining copies of the manifest accompany the waste shipment.

3. Ashland must receive a copy of the manifest signed by the owner or operator of the designated treatement/disposal facility within 35 days of the date the waste was accepted by the first transporter. If Ashland does not receive this copy, we will contact the transporter and/or the owner/operator of the designated TSD facility to determine the status of the waste.

   If Ashland cannot trace the whereabouts of the waste and get a copy of the signed manifest within 10 additional days (45 days total from shipment), Ashland must file an Exception Report to the EPA Regional Administrator.

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PROPER DOT SHIPPING NAME "Shipping Name/Hazard Class"
(Same info as on the manifest)

HAZARDOUS WASTE

FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE, OR PUBLIC SAFETY AUTHORITY, OR THE U.S. ENVIRONMENTAL PROTECTION AGENCY

GENERATOR INFORMATION.

NAME "Generator Name" on manifest

ADDRESS "Generator Address" on manifest

CITY _____ STATE _____ ZIP 00000

EPA ID NO. "Generator EPA I.D. No." EPA WASTE NO. "Hazard I.D. No."

ACCUMULATION START DATE Generator Provides Date MANIFEST DOCUMENT NO. "000886"

HANDLE WITH CARE!

CONTAINS HAZARDOUS OR TOXIC WASTES

Figure 1. Hazardous Waste Label

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SUMMARY REPORT

Incidents requiring the use of the Contingency Plan

Plant Location _____

EPA I.D. # _____

Person Completing Form _____

Date of Incident	Incident	Remedial Action Taken

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Chemical Waste Management Inc.

CHEMICAL WASTE MANAGEMENT, INC.

GUIDELINES FOR PREPARATION OF HAZARDOUS WASTE MANIFESTS

September, 1984

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HAZARDOUS WASTE MANIFESTS

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INTRODUCTION

After several years of deliberation, final uniform hazardous waste manifest regulations have been promulgated by the EPA and D.O.T. Published in the March 20, 1984 Federal Register, the manifest is mandatory for all regulated, off-site shipments of hazardous waste effective September 20, 1984.

DISCLAIMER FOR USE

Chemical Waste Management, Inc. has prepared these Guidelines for the use of its customers and other parties interested in the management of hazardous or toxic waste.

The legislation, regulations, and enforcement policy on which these Guidelines are based are and can be subject to frequent change or interpretation. The user should and is urged to check current legislation, regulations, or enforcement policies of the various federal, state, or local bodies responsible for enforcement of hazardous waste manifest preparation.

Chemical Waste Management assumes no responsibility or liability for the use or the results of the use of these Guidelines by the user.

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Please print or type (Form designed for use on elite (12-pitch) typewriter.)

Form Approved. OMB No. 2000-0404. Expires 7-31-86

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No.

Manifest Document No.

2. Page 1 of

Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address

4. Generator's Phone ( )

A. State Manifest Document Number

B. State Generator's ID

5. Tranporter 1 Company Name

6. US EPA ID Number

C. State Transporter's ID

D. Transporter's Phone

7. Transporter 2 Company Name

8. US EPA ID Number

E. State Transporter's ID

F. Transporter's Phone

9. Designated Facility Name and Site Address

10. US EPA ID Number

G. State Facility's ID

H. Facility's Phone

11. US DOT Description (Including Proper Shipping Name, Hazard Class, and ID Number)	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol	I. State Waste No.
a.					
b.					
c.					
d.					

J. Additional Descriptions for Materials Listed Above

K. Handling Codes for Wastes Listed Above

15. Special Handling Instructions and Additional Information

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national governmental regulations.

Printed/Typed Name | Signature | Date: Month Day Year

GENERATOR

17. Transporter 1 Acknowledgement of Receipt of Materials

Printed/Typed Name | Signature | Date: Month Day Year

18. Transporter 2 Acknowledgement or Receipt of Materials

Printed/Typed Name | Signature | Date: Month Day Year

TRANSPORTER

19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.

Printed/Typed Name | Signature | Date: Month Day Year

FACILITY

EPA Form 8700-22 (3-84)

GENERATOR'S COPY

7-BLS-C6

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Please print or type (Form designed for use on elite (12 pitch) typewriter.)

Form Approved OMB No 2000-0404 Expires 7-31-85

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No. T N D 0 0 0 0 0 1 2 3 4 5 — Manifest Document No 0 0 0 0 1

2. Page 1 of 1

Information in the shaded areas is not required by Federal law.

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3. Generator's Name and Mailing Address
Tennessee Processing Company, Inc.
123 Main Street
Knoxville, Tennessee 37203

4. Generator's Phone (615) 555-1234

A. State Manifest Document Number

B. State Generator's ID

5. Tranporter 1 Company Name
Chemical Waste Management, Inc.

6. US EPA ID Number A L D 0 0 0 6 7 2 4 6 4

C. State Transporter's ID

D. Transporter's Phone

7. Transporter 2 Company Name

8. US EPA ID Number

E. State Transporter's ID

F. Transporter's Phone

9. Designated Facility Name and Site Address
Chemical Waste Management, Inc.
Rt. 17 Milepost 163
Emelle, Alabama 35499

10. US EPA ID Number A L D 0 0 0 8 2 2 5 6 4

G. State Facility's ID

H. Facility's Phone

11. US DOT Description (Including Proper Shipping Name, Hazard Class, and ID Number)	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol	I. Waste No.
a. Waste Hydrofluoric Acid Solution Corrosive Material, UN 1790 CWM Profile # COL A 12345	001	TT	3000	G	D002
b.					
c.					
d.					

J. Additional Descriptions for Materials Listed Above
Liquid, density 1.2

K. Handling Codes for Wastes Listed Above

15. Special Handling Instructions and Additional Information
Placard "Corrosive"

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national governmental regulations.

Printed/Typed Name: John Jones — Signature: — Date: Month 09 Day 20 Year 84

17. Transporter 1 Acknowledgement of Receipt of Materials

Printed/Typed Name: Robert Smith — Signature: — Date: Month 09 Day 20 Year 84

18. Transporter 2 Acknowledgement of Receipt of Materials

Printed/Typed Name: — Signature: — Date: Month . Day . Year .

19. Discrepancy Indication Space

20. Facility Owner or Operator. Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.

Printed/Typed Name: Richard Adams — Signature: — Date: Month 09 Day 20 Year 84

GENERATOR — TRANSPORTER — FACILITY

EPA Form 8700-22 (3-84)

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ORIGINAL — RETURN TO GENERATOR

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MANIFEST AQUISITION

The U.S. EPA has established a hierarchy of aquisition for generators to follow in determining where to obtain blank manifest forms. The specific regulations are found in 40 CFR 262.21.

262.21 Acquisition of Manifests

(a) If the State to which the shipment is manifested (consignment State) supplies the Manifest and requires its use, then the generator must use that Manifest.

(b) If the consignment State does not supply the Manifest, but the State in which the generator is located (generator State) supplies the Manifest and requires its use, then the generator must use that State's Manifest.

(c) If neither the generator State or the consignment State supplies the Manifest, then the generator may obtain the Manifest from any source.

The states have developed various programs to deal with manifest aquisition. They can be placed into the following categories.

I. States with no aquisition requirements.

The following states have choosen not to supply manifests to generators of hazardous waste.

Alaska	Kentucky	Oregon
Arizona	Mississippi	South Dakota
Colorado	Montanta	Tennessee
District of Columbia	Nebraska	Utah
Florida	Nevada	Virginia
Hawaii	New Mexico	Washington
Idaho	North Carolina	West Virginia
Indiana	North Dakota	Wyoming
Iowa	Ohio	
Kansas		

II. States that will supply the manifest based on the hierarchy of aquisition established.

The following states will supply their printing of the manifest and require it's use for all shipments destined to their state or which originate in their state and are destined to a state without manifest aquisition requirements.

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Alabama
Arkansas
California
Connecticut
Delaware
Georgia
Illinois
Louisiana
Maine
Maryland
Massachusetts
Michigan
Missouri
New Hampshire
New Jersey
New York
Oklahoma
Pennsylvania
Rhode Island
South Carolina
Texas
Vermont
Wisconsin

The following matrix can be used to determine where to obtain the form.

MANIFEST AQUISITION MATRIX

DISPOSAL STATE

ORIGIN STATE	ALABAMA	ARIZONA	CALIF	FLORIDA	ILLINOIS	LOUISIANA	MASS	OHIO	OREGON	TEXAS	WISCONSIN
ALABAMA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
ALASKA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
ARIZONA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
ARKANSAS	AL via TSDF	AR	AR	AR	IL	LA	MA	AR	AR	TX	WI
CALIFORNIA	AL via TSDF	CA	CA	CA	IL	LA	MA	CA	CA	TX	WI
COLORADO	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
CONNECTICUT	AL via TSDF	CT	CT	CT	IL	LA	MA	Any Source	Any Source	TX	WI
DELAWARE	AL via TSDF	DE	DE	DE	IL	LA	MA	DE	DE	TX	WI
DIST OF COLUMBIA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
FLORIDA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
GEORGIA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
HAWAII	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
IDAHO	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
ILLINOIS	AL via TSDF	IL	IL	IL	IL	LA	MA	IL	IL	TX	WI
INDIANA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI

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MANIFEST AQUISITION MATRIX

DISPOSAL STATE

ORIGIN STATE	ALABAMA	ARIZONA	CALIF	FLORIDA	ILLINOIS	LOUISIANA	MASS	OHIO	OREGON	TEXAS	WISCONSIN
IOWA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
KANSAS	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
KENTUCKY	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
LOUISIANA	AL via TSDF	LA	LA	LA	IL	LA	MA	LA	LA	TX	WI
MAINE	AL via TSDF	ME	ME	ME	IL	LA	MA	ME	ME	TX	WI
MARYLAND	AL via TSDF	MD	MD	MD	IL	LA	MA	MD	MD	TX	WI
MASSACHUSETTS	AL via TSDF	MA	MA	MA	IL	LA	MA	MA	MA	TX	WI
MICHIGAN	AL via TSDF	MI	MI	MI	IL	LA	MA	MI	MI	TX	WI
MINNESOTA	AL via TSDF				IL	LA	MA			TX	WI
MISSISSIPPI	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
MISSOURI	AL via TSDF	MO	MO	MO	IL	LA	MA	MO	MO	TX	WI
MONTANA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
NEBRASKA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
NEVADA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
NEW HAMPSHIRE	AL via TSDF	NH	NH	NH	IL	LA	MA	NH	NH	TX	WI

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MANIFEST AQUISITION MATRIX

DISPOSAL STATE

ORIGIN STATE	ALABAMA	ARIZONA	CALIF	FLORIDA	ILLINOIS	LOUISIANA	MASS	OHIO	OREGON	TEXAS	WISCONSIN
NEW JERSEY	AL via TSDF	NJ	NJ	NJ	IL	LA	MA	NJ	NJ	TX	WI
NEW MEXICO	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
NEW YORK	AL via TSDF	NY	NY	NY	IL	LA	MA	NY	NY	TX	WI
NORTH CAROLINA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
NORTH DAKOTA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
OHIO	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
OKLAHOMA	AL via TSDF	OK	OK	OK	IL	LA	MA	OK	OK	TX	WI
OREGON	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
PENNSYLVANIA	AL via TSDF	PA	PA	PA	IL	LA	MA	PA	PA	TX	WI
RHODE ISLAND	AL via TSDF	RI	RI	RI	IL	LA	MA	RI	RI	TX	WI
SOUTH CAROLINA	AL via TSDF	SC	SC	SC	IL	LA	MA	SC	SC	TX	WI
SOUTH DAKOTA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
TENNESSEE	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
TEXAS	AL via TSDF	TX	TX	TX	IL	LA	MA	TX	TX	TX	WI
UTAH	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI

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MANIFEST AQUISITION MATRIX

DISPOSAL STATE

ORIGIN STATE	ALABAMA	ARIZONA	CALIF	FLORIDA	ILLINOIS	LOUISIANA	MASS	OHIO	OREGON	TEXAS	WISCONSIN
VERMONT	AL via TSDF	VT	VT	VT	IL	LA	MA	VT	VT	TX	WI
VIRGINIA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
WASHINGTON	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
WEST VIRGINIA	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI
WISCONSIN	AL via TSDF	WI	WI	WI	IL	LA	MA	WI	WI	TX	WI
WYOMING	AL via TSDF	Any Source	Any Source	Any Source	IL	LA	MA	Any Source	Any Source	TX	WI

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MANIFEST PREPARATION FEDERAL REQUIREMENTS

The uniform manifest contains 20 items of required information. Each item must be complete and correct. Items 1-16 are to be completed by the generator, items 17-18 by the transporter(s), and items 19-20 by the treatment, storage or disposal facility. Care must be taken to assure that all entries are complete and accurate. All copies must be legible.

The following are the specific requirements of each item.

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GENERATOR REQUIREMENTS

UNIFORM HAZARDOUS WASTE MANIFEST	1. Generator's US EPA ID No. T·N·D·0·0·0·0·1·2·3·4·5	Manifest Document No. 0·0·0·0·1	2. Page 1 of 1
3. Generator's Name and Mailing Address Tennessee Processing Company, Inc. 123 Main Street Knoxville, Tennessee 37203 4. Generator's Phone (615) 555-1234			

Item 1. Generator's U.S. EPA ID Number - Manifest Document Number

Enter the generator's U.S. EPA twelve digit identification number and on annually unique five digit number assigned to this Manifest (e.g., 00001) by the generator.

Item 2. Page 1 of _____

Enter the total number of pages used to complete this Manifest, including the number of Continuation Sheets, if any. (i.e., 1 of 2, 1 of 3)

Item 3. Generator's Name and Mailing Address

Enter the name and mailing address of the generator. The address must be the location that will manage the returned Manifest forms.

Item 4. Generator's Phone Number

Enter a telephone number where an authorized agent of the generator may be reached in the event of an emergency.

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5. Tranporter 1 Company Name CHEMICAL WASTE MANAGEMENT, INC.	6. US EPA ID Number A·L·D·0·0·0·6·7·2·4·6·4
7. Transporter 2 Company Name	8. US EPA ID Number · · · · · · · · · · · ·
9. Designated Facility Name and Site Address CHEMICAL WASTE MANAGEMENT, INC. RT. 17 MILEPOST 163 EMELLE, ALABAMA 35499	10. US EPA ID Number A·L·D·0·0·0·8·2·2·4·6·4

Item 5. Transporter 1 Company Name

Enter the company name of the first transporter who will transport the waste.

Item 6. U.S. EPA ID Number

Enter the U.S. EPA twelve digit identification number of the first transporter identified in item 5.

Item 7. Transporter 2 Company Name

Enter the company name of the second transporter. If more than two transporters are used to transport the waste, use a Continuation Sheet(s) and list the transporters in the order they will be transporting the waste.

Item 8. U.S. EPA ID Number

Enter the U.S. EPA twelve digit identification number of the second transporter identified in item 7.

Item 9. Designated Facility Name and Site Address

Enter the company name and site address of the facility designated to receive the waste listed on this Manifest. The address must be the site address.

Item 10. U.S. EPA ID Number

Enter the U.S. EPA twelve digit identification number of the designated facility identified in item 9.

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11. US DOT Description (Including Proper Shipping Name, Hazard Class, and ID Number)	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol
a. Waste Hydrofluoric Acid Solution Corrosive Material, UN 1790 CWM Profile # COL A 12345	0·0·1	T·T	·3·0·0·0	G
b.				

Item 11. U.S. DOT Description (Including Proper Shipping Name, Hazard Class, and ID Number [UN/NA])

Enter the U.S. DOT Proper Shipping Name, Hazard Class, and ID Number (UN/NA) for each waste as identified in 49 CFR 172.101

CWM requires the profile number after each shipping description, i.e., "CWM Profile # COL-A-12345".

Note: - If additional space is needed for waste descriptions, enter these additional descriptions in item 28 on the Continuation Sheet.

Item 12. Containers (No. and Type)

Enter the number of containers for each waste and the appropriate abbreviation from Table I (below) for the type of container.

Table I -- Types of Containers

DM = Metal drums, barrels, kegs
DW = Wooden drums, barrels, kegs
DF = Fiberboard or plastic drums, barrels, kegs
TP = Tanks portable
TT = Cargo tanks (tank trucks)
TC = Tank cars
DT = Dump truck
CY = Cylinders
CM = Metal boxes, cartons, cases (including roll-offs)
CW = Wooden boxes, cartons, cases
CF = Fiber or plastic boxes, cartons, cases
BA = Burlap, cloth, paper or plastic bags

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Item 13. Total Quantity

Enter the total quantity of waste described on each line. This quantity should not be an estimate, but should be as precise as possible.

Note: Some states have a specific requirement note state section.

Item 14. Unit (Wt./Vol.)

Enter the appropriate abbreviation from Table II (below) for the unit of measure.

Table II - Units of Measure

G = Gallons (liquids only)
P = Pounds
T = Tons (2000 lbs)
Y = Cubic yards
L = Liters (liquids only)
K = Kilograms
M = Metric tons (1000 kg)
N = Cubic meters

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J. Additional Descriptions for Materials Listed Above
Liquid, density 1.2

15. Special Handling Instructions and Additional Information
Placard "Corrosive"

Item 15. Special Handling Instructions and Additional Information

If an alternate facility is designated, the information and identification must appear here. Generators may use this space to indicate special transportation, treatment, storage, or disposal information or Bill of Lading information. For international shipments, generators must enter in this space the point of departure (City and State) from the U.S. for those shipments destined for treatment, storage, or disposal outside the jurisdiction of the United States.

Item 16. Generator's Certification

The generator must read, sign (by hand), and date the certification statement. If a mode other than highway is used, the word "highway" should be lined out and the appropriate mode (rail, water, or air) inserted in the space below. If another mode in addition to the highway mode is used, enter the appropriate additional mode (e.g., and rail) in the space below.

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TRANSPORTATION REQUIREMENT

16. **GENERATOR'S CERTIFICATION:** I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national governmental regulations.		Date
Printed/Typed Name John Jones	Signature John Jones	Month Day Year 0 9 2 0 8 4
17. Transporter 1 Acknowledgement of Receipt of Materials		Date
Printed/Typed Name Robert Smith	Signature Robert Smith	Month Day Year 0 9 2 0 8 4
18. Transporter 2 Acknowledgement of Receipt of Materials		Date
Printed/Typed Name	Signature	Month Day Year

Item 17. Transporter 1 Acknowledgement of Receipt of Materials

Enter the name of the person accepting the waste on behalf of the first transporter. That person must acknowledge acceptance of the waste described on the Manifest by signing and entering the date of receipt.

Item 18. Transporter 2 Acknowledgement of Receipt of Materials

Enter, if applicable, the name of the person accepting the waste on behalf of the second transporter. The person must acknowledge acceptance of the waste described on the Manifest by signing and entering the date of receipt.

Note: - International Shipments - Transporter Responsibilities.

Exports - Transporters must sign and enter the date the waste left the United States in item 15 of Form.

Imports - Shipments of hazardous waste regulated by RCRA and transported into the United States from another country must upon entry be accompanied by the U.S. EPA Uniform Hazardous Waste Manifest. Transporters who transport hazardous waste into the United States from another country are responsible for completing the Manifest (40 CFR 263.10(c)(1)).

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TSD FACILITY REQUIREMENTS

19. Discrepancy Indication Space		
20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.		Date
Printed/Typed Name Richard Adams	Signature Richard Adams	Month Day Year 0 9 2 0 8 4

Item 19. Discrepancy Indication Space

The authorized representative of the designated (or alternate) facility's owner or operator must note in this space any significant discrepancy between the waste described on the Manifest and the waste actually received at the facility.

Owners and operators of facilities located in unauthorized States (i.e., the U.S. EPA administers the hazardous waste management program) who cannot resolve significant discrepancies within 15 days of receiving the waste must submit to their Regional Administrator a letter with a copy of the Manifest at issue describing the discrepancy and attempts to reconcile it (40 CFR 264.72 and 265.72).

Owners and operators of facilities located in authorized States (i.e., those States that have received authorization from the U.S. EPA to administer the hazardous waste program) should contact their State agency for information on State Discrepancy Report requirements.

Item 20. Facility Owner or Operator: Certification of Receipt of Hazardous Materials Covered by This Manifest Except as Noted in Item 19

Print or type the name of the person accepting the waste on behalf of the owner or operator of the facility. That person must acknowledge acceptance of the waste described on the Manifest by signing and entering the date of receipt.

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ADDITIONAL CWM REQUIREMENTS

1. CWM requires that the profile number be shown after each waste description in item 11.

2. CWM requires the physical state of the material to be shown in item J. When a manifest describes multiple streams, the above information must be related to the description by identifying the entry line (i.e., 11a - 11b, etc).

3. CWM requests that all printed names of persons signing the manifest include that person's title.

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CONTINUATION SHEET PREPARATION

The Uniform Manifest Continuation Sheet (U.S. EPA Form 8700-22A) must be used if:

More than two transporters are to be used to transport the waste, or;

More space is required for the U.S. DOT description and related information in Item 11 of the Manifest.

The item by item preparation of the Continuation Sheet can be accomplished as follows:

Item 21. Generator's U.S. EPA ID Number - Manifest Document Number

Enter the generator's U.S. EPA twelve digit identification number and the unique five digit number assigned to this Manifest (e.g., 00001) as it appears in item 1 on the first page of the Manifest.

Item 22. Page

Enter the page number of this Continuation Sheet.

Item 23. Generator's Name

Enter the generator's name as it appears in item 3 on the first page of the Manifest.

Item 24. Transporter Company Name

If additional transporters are used to transport the waste described on this Manifest, enter the company name of each additional transporter in the order in which they will transport the waste. Enter after the word "Transporter" the order of the transporter. For example, Transporter 3 Company Name. Each Continuation Sheet will record the names of two additional transporters.

Item 25. U.S. EPA ID Number

Enter the U.S. EPA twelve digit identification number of the transporter described in item 24.

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Item 26. Transporter Company Name

If additional transporters are used to transport the waste described on this Manifest, enter the company name of each additional transporter in the order in which they will transport the waste. Enter after the word "Transporter" the order of the transporter. For example, Transporter 4 Company Name. Each Continuation Sheet will record the names of two additional transporters.

Item 27. U.S. EPA ID Number

Enter the U.S. EPA twelve digit identification number of the transporter described in item 26.

Item 28. U.S. DOT Description Including Proper Shipping Name, Hazardous Class, and ID Number (UN/NA)

Refer to item 11.

Item 29. Containers (No. and Type)

Refer to item 12.

Item 30. Total Quantity

Refer to item 13.

Item 31. Unit (Wt./Vol.)

Refer to item 14.

Item 32. Special Handling Instructions

Generators may use this space to indicate special transportation, treatment, storage, or disposal information or Bill of Lading information. State are not authorized to require additional, new, or different information in this space.

TRANSPORTERS

Item 33. Transporter Acknowledgement of Receipt of Materials

Enter the same number of the Transporter as identified in item 24. Enter also the name of the person accepting the waste on behalf of the Transporter (Company Name) identified in item 24. That person must acknowledge acceptance of the waste

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described on the Manifest by signing and entering the date of receipt.

Item 34. Transporter Acknowledgement of Receipt of Materials

Enter the same number as identified in item 26. Enter also the name of the person accepting the waste on behalf of the Transporter (Company Name) identified in item 26. That person must acknowledge acceptance of the waste described on the Manifest by signing and entering the date of receipt.

Owners and Operators of Treatment, Storage, or Disposal Facilities

Item 35. Descrepancy Indication Space

Refer to item 19.

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OPTIONAL STATE REQUIREMENTS

The uniform hazardous waste manifest contains eleven items of optional information which the origin and destination state may require. These items are shaded on the manifest and lettered A thru K.

These requirements vary from state to state. Generators should contact the origin and destination state authorities for specific information on their requirements.

Attached are summary sheets for each state which can be used as a guideline to meeting the state requirements.

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OHIO

Agency

Ohio Environmental Protection Agency
Division of Hazardous Materials
361 E. Broad Street
Columbus, Ohio 43215

Aquisition of Manifests

Ohio will not supply generators with a copy of the manifest.

State Required Additional Information

The following additional information will be required on manifests describing waste which originates in or is destined to Ohio:

1. U.S. EPA waste number - Item I.

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QUALIFICATION STATEMENTS

Jerry W.Boone, Research Services Coordinator has had 6 years experience as a Research Services Coordinator directing R&D safety, operational and hazardous waste activities. Prior to this position, Mr. Boone was a Chemist with the Industrial Chemicals & Solvents Division for 6 years and worked in the Product Applications Development Laboratory in Ashland, Kentucky for 6 years as a chemist. He attends training seminars which concern DOT, RCRA, etc. regulations and other related activities. All RCRA training is reported at least annually.

James A. Mills has been Safety Coordinator at Ashland for three years. Previously he was Fire Chief at Dublin, Ohio and Norton, Ohio, and has been active in the Fire Service for 17 years. Mr. Mills also worked in Production Management at the Hoover Company for 11 years.

He is a certified Paramedic in Ohio and has attended numerous Hazardous Materials courses, seminars, and schools. He is a certified Instructor in Ohio for Basic Fire Service Training, In Service Fire Service Training, Fire Safety Inspector, Fire Brigades and Rapid Water Rescue, and has taught at the Ohio Fire Academy.

Mr. Mills holds an A.A.S. in Fire Science.

Arlene A. Hendrickson, has had nine years experience as an environmental engineer for Ashland Chemical Company. Has had extensive training in RCRA related topics. Responsible for the development of the RCRA audio-visual training program and has been involved in training seminars conducted for Ashland Chemical Company personnel.

Ms. Hendrickson has a B.S. in chemical engineering, one year in graduate work in chemistry and an M.B.A. degree.

David L. Anderson, Environmental Engineer, has had four years experience has an environmental engineer for Ashland Chemical Company. Total engineering experience both as a chemical and environmental engineer has been 12 years. Has attended several RCRA related courses and has been involved in training seminars conducted for Ashland Chemical Company personnel.

Mr. Anderson has a B.S. and M.S. in chemical engineering.

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Ref: Sec. 264.16 and 122.25(a)(12)

PERSONNEL TRAINING

Ashland's Research & Development Laboratory has an extensive safety program which includes training required by the Resource Conservation and Recovery Act (RCRA) and Oil Pollution Prevention Regulations (SPCC Plan). Each employee attends monthly safety meetings. Employees in the Chemical Pilot Plant have safety meetings weekly. In addition to the safety meetings, each employee is provided with the R&D Safety Manual. The safety manual contains company safety regulations and provides basic instructions on how to handle chemical products at the Lab. Periodic training is provided to reinforce safety manual procedures and tests are given to insure that the procedures are understood. In addition to this there is on-th-job training covering the handling of hazardous chemicals. Hazardous wastes would be handled in the samemanner as hazardoius chemicals. A combination of the safety manual, safety meetings and on-the-job training ensures that Ashland personnel meet the standards under RCRA. R&D personnel must not work in unsupervised positions until they have completed this training.

An audio visual training program on RCRA will be used for the annual review of RCRA.

Employees involved in handling hazardous chemical products and wastes are required to take the following additional training:

1. Forklift operators must successfully complete training before being allowed to operate the unit.

2. Plant handlers of hazardous chemical products and wastes receive on-the-job training from the lab supervisor and experienced lab personnel. Part of the on-the-job training includes instructions on properly preparing manifests, placarding drums, etc., for hazardous chemical products and wastes.

3. An audio-visual training program on RCRA is given to lab personnel (See Attachment I). (SLIDE presentation)

4. Procedures for use, inspection (see "Inspection Schedule and Log" in Contingency Plan), repair, and replacement of facility emergency equipment, monitoring equipment and alarm system.

5. Employee requirements due to a fire, explosion or groundwater contamination incidents. These requirements include proper usage of fire extinguishers, employee protection, proper reporting procedures, etc.

6. Shutdown or securing lab operations during a fire, explosion, power blackout, pump failure, operational spillage, etc.

The specific requirements addressed in Training Items 1-6 must be successfully completed before a new employee is allowed to work in an unsupervised area of the plant. The training oulined in Attachment II on page H-40 will be completed within 6 months of an employee being hired into the position. The requirements associated with Training Items 1-6 are repeated at least annually, and certain areas are reinforced to insure continuous understanding at monthly safety meetings. The specifics of training for each job title are contained in Attachment II. Records of all personnel training are maintained at the lab.

Hazardous wastes as defined by RCRA are handled in the same manner as hazardous chemical products. As a result, the combination of safety manuals, safety meetings, tests and on-the-job training sessions ensure that Ashland IC&S personnel meet the standards under RCRA.

Files of all safety and RCRA related training are maintained by the Research Services Coordinator. The key personnel responsible for employee safety training with special emphasis upon RCRA are listed below along with major responsibilities:

Each person listed here is responsbile for reading the R&D Safety Manual with special emphasis on the waste disposal section. Each employee will attend the appropriate monthly/weekly safety meeting. The Research Manager and Research Services Coordinator must keep a copy of the RCRA Contingency Plan and Emergency Procedures and be familiar with the information contained therein. Annually all employees listed here must view an audio visual training program on RCRA or review the waste management aspects of their jobs at a safety meeting.

Attendance records are kept for all safety meetings. This will serve as documentation for training received under RCRA.

Job title for each position at the laboratory related to hazardous waste management and the name of the employee filling each job is as follows:

Research Manager	Michel E. Mullier
Research Services Coordinator	Jerry W. Boone
Shipping Receiving Clerk	James W. Miketa
Chemical Packaging Clerk	Opal Hunter
Pilot Plant	See Attached Sheets

Job descriptions for each of these jobs is attached.

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M. E. MULLIER

Research Manager: Responsible for:

1) Analytical Dept.: All analyses, including air monitoring analyses for the Environmental group.

2) Research Services:

   a) Packaging and shipping according to federal rules and regulations

   b) Receiving

   c) Stockroom

   d) Waste disposal

   e) Laboratory modifications and improvements

3) Safety in R&D Building

4) Coordination with Building Services

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MEMORANDUM

To: J. Boone

Date: March 7, 1977

From: M. E. Mullier

cc: J. E. Lewis
E. F. Dalton
C. E. Corn

Subject: Research Services Coordinator

As per our discussion of this morning, I have summarized here what I feel will be your main responsibilities for the foreseeable future:

Safety

- Act as permanent secretary to the Safety Committee
- After reviewing with M. E. Mullier, implement the recommendations of the Safety Committee
- Maintain records for OSHA/Ashland safety requirements and follow-up on these matters

Security

- Maintain Security files
- Coordinate security matters through M. E. Mullier

Services

- Supervise glassware washing activities
- Supervise stockroom operations
- Supervise solvent storage room
- Supervise removal of waste solvent from the laboratories
- Supervise delivery of packages to appropriate laboratory people
- Administer the tax-free alcohol requirements
- Help R&D personnel when research services problems occur
- Coordinate R&D needs with Building Services

I realize that this is a very heavy program but I feel confident that, together, we can handle it.

M E Mullier

MEM/re

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April 23, 1979

Job Duties of Shipping-Receiving Clerk

1. Maintain an alphanumerical filing system of all current and past purchase orders.

2. Match packing slips with purchase orders, fill out Receipt of Materials form, send copies of purchase order, packing slip and Receipt of Materials form to Accounts Payable, attach copy of Receipt of Materials form to purchase order and file in R & D receiving office.

3. Receive and unload trucks at the R & D Receiving Dock, and request assistance from the delivery clerk with the items if too many, large, or bulky.

4. Maintain work area in a clean, neat, orderly, safe and secure manner.

5. Prepare Bill of Lading for all out-bound surface freight shipments in compliance with D.O.T. regulations and route out-bound surface shipments.

6. Maintain files of all incoming and out-bound Bill of Lading for surface shipments and air bills for air freight.

7. Receive and route any incoming calls for lights, spills, trash pickup, etc., to Building Services personnel.

8. Maintain records on all movements of specialty gases.

9. Check liquid nitrogen tank daily and call for drop shipments if needed.

10. Inspect trucks and attach placards if needed to comply with D.O.T. regulations.

11. Trace any lost shipments through surface freight companies.

12. Be responsive to request from Research Services Coordinator.

13. Assist delivery clerk by identifying and marking all packages which the receipient cannot be identified by the address label or packing slip.

14. Maintain files of sample request forms for all incoming samples for R & D employees.

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April 25, 1979

JOB DESCRIPTION OF CHEMICAL PACKAGING CLERK

1. Package and label all chemicals to be shipped in accordance with D.O.T. and IATA regulations.

2. Sign Restricted Article Certifications and prepares shippers letters of instructions and commercial invoice.

3. Maintains and keeps up-to-date all regulations including D.O.T. Title 49, IATA, Ashland freight classifications and the QA Master list pertaining to the handling of hazardous materials.

4. Responsible for ensuring compliance of all chemical shipments with applicable regulations.

5. Verifies all freight bills to find erroneous and/or duplicate payments.

6. Maintains records of all shipments and trace shipments.

7. Maintains liaison with carriers including selection for individual shipments with regard to rates, service, etc.

8. Maintain an adequate inventory of proper packaging materials to meet current regulations.

9. Maintain service contracts with scale and postage machine companies.

10. Obtain packing exceptions from packaging engineering for shipments.

11. Obtain proper shipping name and freight classification from Traffic Department and hazards classification from EOSD when they are not specifically listed in the Ashland Freight Classification book or QA Master list.

12. Obtain necessary approvals of appropriate management for air shipments.

13. Take mandatory air and surface tests, and make perfect scores on both on a yearly basis.

14. Approve sample requests to ship materials out of the Dublin complex.

15. Make out bills of lading for all outbound shipments and confer with the Traffic Department on freight classifications.

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CHEMICAL PILOT PLANT STAFF

Gary W. Felton - Group Leader

Lora Polk - Secretary

Sr. Research Engineers

M. Scott Baker
Robert D. Fairchild

Research Engineer

Scott Simmers

Staff Lab Technician

Vincent S. Williams

Lab Technician

Kent C. Gay
W. Keith Hunter
John W. Martin, Jr.
John T. Bennett

7957
JOB DESCRIPTION QUESTIONNAIRE
ASHLAND OIL, INC.

PLEASE TYPE:

OCT 26 1984
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I. IDENTIFICATION INFORMATION

JOB TITLE: GROUP LEADER

NAME: G.P. ANDERSON H-9

REPORTS TO: R.V. NORTON

TITLE: RESEARCH MANAGER

COMPANY/DIVISION: ASHLAND CHEMICAL

DEPARTMENT/SECTION: VENTURE R+D

LOCATION: DUBLIN OHIO

DATE PREPARED: 10/5/89

II. BASIC FUNCTIONS

THIS POSITION IS ACCOUNTABLE FOR (SUMMARIZE)

GENERATION AND INTERPRETATION OF ENGINEERING DATA ASSOCIATED WITH PILOT PLANT ACTIVITIES IN SUPPORT OF ASHLAND CHEMICAL VENTURE.

THIS FUNCTION INCLUDES THE FOLLOWING SPECIFIC ACTIVITIES OR RESPONSIBILITIES AND/OR CHARACTERISTIC PROJECTS: (LIST AND BRIEFLY DESCRIBE AT LEAST THREE OR MORE OF THE MOST SIGNIFICANT USING ADDITIONAL SHEET IF NECESSARY)

- DEVELOPMENT OF EXPERIMENTAL PROGRAMS TO OBTAIN HEAT AND MATERIAL BALANCES FOR NEW PRODUCTS BEFORE SCALE UP TO COMMERCIAL LEVELS.

REVIEW AND APPROVAL OF PILOT PROCEDURES USED TO GUIDE DATA COLLECTION

- INTERACTION WITH OTHER GROUPS TO EXPEDITE NEW PRODUCT DEVELOPMENT AS WELL PROVIDING SAMPLES OF EXISTING PRODUCTS TO POTENTIAL CUSTOMERS

III. EDUCATION AND EXPERIENCE REQUIREMENTS

CONSIDER ONLY THE LEVEL REQUIRED TO PERFORM YOUR JOB. (DO NOT INDICATE YOUR OWN UNLESS YOU FEEL IT SHOULD BE THE MINIMUM REQUIRED.)

CHECK ONE:

1 [ ] HIGH SCHOOL
2 [ ] HIGH SCHOOL PLUS
3 [x] BACHELORS DEGREE
4 [x] MASTERS DEGREE
5 [ ] PH. D
6 [ ] OTHER

IF TWO THRU SIX ARE CHECKED, PLEASE INDICATE ACADEMIC DISCIPLINE OR FIELD OF STUDY, ETC.

CHEM. ENG. CHEMISTRY, MECHANICAL ENGINEERING

WHAT, IF ANY, ADDITIONAL RELATED EXPERIENCE IS REQUIRED TO FULFILL THE REQUIREMENTS OF THIS JOB? (DO NOT INDICATE YOUR OWN PREVIOUS EXPERIENCE UNLESS YOU FEEL IT SHOULD BE THE MINIMUM REQUIRED.)

PLEASE INDICATE THE LENGTH OF RELATED EXPERIENCE. THIS SHOULD BE EXPRESSED AS YEARS OF BROAD AND VARIED EXPERIENCE RATHER THAN THE SAME EXPERIENCE YEAR AFTER YEAR. (CHECK ONE)

[ ] LESS THAN 1 YEAR
[ ] 1 - 3 YEARS
[x] 3 - 5 YEARS
[ ] 5 - 10 YEARS
[ ] 10 YEARS PLUS

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7957
JOB DESCRIPTION QUESTIONNAIRE
ASHLAND OIL, INC.

PLEASE TYPE:

I. IDENTIFICATION INFORMATION

JOB TITLE:

NAME:

REPORTS TO: G. P. Anderson

TITLE: Group Leader - Chemical Pilot Plant

COMPANY/DIVISION: Ashland Chemical Company

DEPARTMENT/SECTION: Venture Chemical Pilot Plant - R&D

LOCATION: Dublin, Ohio

DATE PREPARED: 10/08/81

THIS POSITION IS ACCOUNTABLE FOR (SUMMARIZE)

Secretary to Group Leader, 4 Research Engineers, 13 Technicians

II. BASIC FUNCTIONS

THIS FUNCTION INCLUDES THE FOLLOWING SPECIFIC ACTIVITIES OR RESPONSIBILITIES AND/OR CHARACTERISTIC PROJECTS: (LIST AND BRIEFLY DESCRIBE AT LEAST THREE OR MORE OF THE MOST SIGNIFICANT USING ADDITIONAL SHEET IF NECESSARY)

General Office - Sorting, opening, and distributing mail, answer phones, correspondence, filing (correspondence, orders, literature, floor plans, etc.), time sheets and attendance charts, supplies, maintaining logs on runs, incoming/outgoing correspondence, procedures, safety data, technical, mechanical and confidential information, company truck, forklift, etc. Typing consisting of monthly/quarterly reports, purchase orders, sample requisitions various forms, procedures, misc. memos, (etc.) Creat necessary forms for analysis of products, operation checklists, etc. Ordering & Logging material & equipment necessary for plant operation, expediting & vendor contact. Work with Receiving/Shipping to coordinate receivables, storage and follow up paperwork. Coordinating shipping papers for equipment repairs, sample for analysis & samples to customers, etc. Inventory Control Log materials & equipment ordered & received. Maintain system for ordering raw materials to keep necessary stock. Scheduling meetings, conference rooms, food, drinks, etc. Schedule airline reservations, rental cars, motel/hotel reservations.

III. EDUCATION AND EXPERIENCE REQUIREMENTS

CONSIDER ONLY THE LEVEL REQUIRED TO PERFORM YOUR JOB. (DO NOT INDICATE YOUR OWN UNLESS YOU FEEL IT SHOULD BE THE MINIMUM REQUIRED.)

CHECK ONE:

1 [ ] HIGH SCHOOL
2 [XX] HIGH SCHOOL PLUS
3 [ ] BACHELORS DEGREE
4 [ ] MASTERS DEGREE
5 [ ] PH. D
6 [ ] OTHER

IF TWO THRU SIX ARE CHECKED, PLEASE INDICATE ACADEMIC DISCIPLINE OR FIELD OF STUDY, ETC.

WHAT, IF ANY, ADDITIONAL RELATED EXPERIENCE IS REQUIRED TO FULFILL THE REQUIREMENTS OF THIS JOB? (DO NOT INDICATE YOUR OWN PREVIOUS EXPERIENCE UNLESS YOU FEEL IT SHOULD BE THE MINIMUM REQUIRED.)

Purchasing & Expediting functions, First Aid Training, Any Safety Programs.

PLEASE INDICATE THE LENGTH OF RELATED EXPERIENCE. THIS SHOULD BE EXPRESSED AS YEARS OF BROAD AND VARIED EXPERIENCE RATHER THAN THE SAME EXPERIENCE YEAR AFTER YEAR. (CHECK ONE)

[ ] LESS THAN 1 YEAR
[ ] 1 - 3 YEARS
[X] 3 - 5 YEARS
[ ] 5 - 10 YEARS
[ ] 10 YEARS PLUS

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ATISA
IPLOYMENT REQUISITION (REPLACEMENT)

DATE SUBMITTED 10/10/1980

ARTMENT - DIVISION - COMPANY	JOB TITLE	NO. REQUIRED
Chemical Pilot Plant - Ashland Chemical Co.	Senior Research Engineer	1

LOCATION	FORCE REPORT CODE		DATE REQUIRED
Dublin, Ohio		☐ Regular ☐ Full-Time ☐ Nonexempt ☐ Temporary ☐ Part-Time ☐ Exempt	Immediately

JOB DESCRIPTION: DESCRIBE THE WORK EMPLOYEE WILL DO. IF PART-TIME, INDICATE SCHEDULE OR HOURS PER WEEK. ATTACH ADDITIONAL SHEET IF NEEDED.

The engineer will be assigned to the Pilot Plant to perform process development work for Venture Research and the divisions. The basic function will be to develop bench scale reactor systems for process development by close coordination and liaison between lab chemists from the various Venture Research groups and engineers. The systems are to be designed and operated by using mathematical modeling, engineering concepts and kinetics from own research/calculations/experience and assistance from other company and non-company personnel. The primary initial task will be for the design and construction coordination of a Pilot Plant suitable for fixed and fluid bed reaction analysis and use.

QUALIFICATIONS REQUIRED, INCLUDING EDUCATION AND EXPERIENCE

The candidate must be a Chemical Engineer graduate with a Ph.D degree. Previous experience to be in the field of reaction engineering, mathematical modeling, kinetics, and the design and operation of pilot plant units including catalytic units. An extensive knowledge of instrumentation application is highly desirable. A strong "mechanical intuition" is also highly desirable. A Chemical Engineering graduate with a M.S. degree and outstanding career credentials would be considered in lieu of a Ph.D. candidate.

This hiring action will not exceed my Staffing Table authorized level for regular employees.

SIGNATURE			DATE
[signature]			10/10/80
APPROVED BY	DATE	APPROVED BY	DATE
[signature] 10/20/80			

INSTRUCTIONS: After the necessary approvals have been obtained, retain the department copy and forward remaining copies to the appropriate Employment Manager, Personnel Department.

INTERNAL/EXTERNAL PLACEMENT

COMMENTS

REQUISITION ASSIGNED TO	DATE

REQUISITION FILLED BY	DATE HIRED

DISTRIBUTION: WHITE - Employment, CANARY - Internal Placement, Ashland, Ky., PINK - Department

EMPLOYMENT REQUISITION

REQ. NO. OCT 26 1984
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DATE SUBMITTED: 1-30-80

DEPARTMENT-DIVISION-COMPANY	JOB TITLE	NO. REQUIRED
Chemical Pilot Plant - Ashland Chemical Co.	Engineer (Entry Level)	1
LOCATION: Dublin, Ohio	[X] REGULAR [X] FULL TIME [ ] NON-EXEMPT [ ] TEMPORARY [ ] PART TIME [ ] EXEMPT	DATE REQUIRED: ASAP

JOB DESCRIPTION: DESCRIBE THE WORK EMPLOYEE WILL DO. IF PART TIME, INDICATE SCHEDULE OR HOURS PER WEEK. ATTACH ADDITIONAL SHEET IF NEEDED.

The entry level Chemical Engineer will design and operate various pilot plant equipment such as batch reactors, continuous units, distillation columns, extraction units, etc. He or she will work under the supervision of Senior Process Development Engineers and in close contact with pilot plant technicians. The normal work schedule is a standard 40 hour week but some overtime and shift work can be expected.

QUALIFICATIONS REQUIRED, INCLUDING EDUCATION AND EXPERIENCE:

The candidate should have a BS in Chemical Engineering from an accredited college. He or she should have a strong mechanical inclination, should be able to conceptualize, design, and lueprint a total unit layout including process, utility, and safety equipment. As a replacement for a Senior Pilot Plant Technician with six years experience he or she should be a "hands on" person, aggressive, and constantly aware of safety.

THIS HIRING ACTION WILL NOT EXCEED MY MANNING TABLE AUTHORIZED LEVEL FOR REGULAR EMPLOYEES.

SIGNED [signature] DATE 1-30-80

APPROVED	DATE	APPROVED	DATE

INSTRUCTIONS: AFTER THE NECESSARY APPROVALS HAVE BEEN RECEIVED, RETAIN THE DEPARTMENT COPY AND FORWARD REMAINING COPIES TO THE APPROPRIATE EMPLOYMENT MANAGER, PERSONNEL DEPARTMENT.

INTERNAL PLACEMENT: COMMENTS	RELEASED BY	DATE

REQUISITION ASSIGNED TO: DATE

REQUISITION FILLED BY: DATE HIRED

COPY 1 – EMPLOYMENT COPY 2 – INTERNAL PLACEMENT COPY 3 – DEPARTMENT

FORM 1718

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716A
EMPLOYMENT REQUISITION

DATE SUBMITTED 9/23/1980

DEPARTMENT - DIVISION - COMPANY	JOB TITLE	NO. REQUIRED
land Chemical/Process/R&D/Venture	Pilot Plant Lab Technician	7

LOCATION	FORCE REPORT CODE		DATE REQUIRED
Dublin, Ohio		☒ Regular ☐ Temporary ☒ Full-Time ☐ Part-Time ☒ Nonexempt ☐ Exempt	ASAP 1980

JOB DESCRIPTION: DESCRIBE THE WORK EMPLOYEE WILL DO. IF PART-TIME, INDICATE SCHEDULE OR HOURS PER WEEK. ATTACH ADDITIONAL SHEET IF NEEDED.

For non-routine pilot plant operations in support of and working directly for a chemical engineer or chemist in the Process Development group. The incumbent will be responsible for monitoring the operation of reactors and carefully reporting data and operating conditions. In addition, the incumbent will perform analytical tasks and prepare tables and graphs of the data accumulated from the reactor. Significant mechanical skills are required for the construction, modification and repair of reactors during normal operation. Some familiarity with complex instrumental techniques would be a distinct advantage as the instrumentation for the analytical procedures is quite complex.

QUALIFICATIONS REQUIRED, INCLUDING EDUCATION AND EXPERIENCE

High school graduate required. A degree in a related science is highly desirable. The applicant is required to have a strong inquisitive attitude and ability for the recording and accurate logging of experimental data in a precise manner. The candidate should have significant college training or experience as a laboratory technician in a medical or other science area. Proper attention to safety matters, and a demonstrated record of thoroughness diligence are required. The noraml work schedule will be the standard 40 hours but rtime and shift work can be expected.

This hiring action will not exceed my Staffing Table authorized level for regular employees.

SIGNATURE			DATE
G P Anderson			9/23/80
APPROVED BY	DATE	APPROVED BY	DATE
Richard V. Morton	9/23/80		

INSTRUCTIONS: After the necessary approvals have been obtained, retain the department copy and forward remaining copies to the appropriate Employment Manager, Personnel Department.

INTERNAL/EXTERNAL PLACEMENT

COMMENTS

REQUISITION ASSIGNED TO	DATE
REQUISITION FILLED BY	DATE HIRED

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ATTACHMENT I

RCRA TRAINING (slide presentations)

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RCRA

Resource Conservation and Recovery Act

PART I

Generators

Transporters

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Slide 1 (Ashland Logo)

Slide 2

THE RESOURCE CONSERVATION

AND RECOVERY ACT

Slide 3

Federal regulations that became effective on November 19, 1980, have put into motion a program to regulate the handling of hazardous wastes from "cradle to grave".

Slide 4

The regulations under the Resource Conservation and Recovery Act or RCRA require an inventory of all businesses in the United States that generate; transport; and treat, store or dispose of hazardous wastes. These regulations impose severe civil and criminal penalties on those who do not comply.

Slide 5

The rules also establish a manifest system which shows who generates waste, where it is going, and whether it gets there safely. In some cases, the federal government has authorized certain states to act on its behalf. Actually, RCRA is designed to encourage each state to act as the primary enforcement agency should they choose to do so.

Slide 6

Ashland is one of many firms that must follow RCRA standards. Not all Ashland locations are involved with every facet of these regulations. As each separate type of activity regulated by RCRA is described in this program, note whether that activity applies to your particular location and what you must do to comply with these standards.

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Slide 7

For the next few minutes, we will present Part I of our total RCRA program. It will concentrate on the responsibilities of generators and transporters as defined by RCRA regulations.

Slide 8

Part II will deal with those facilities that treat, store, or dispose of hazardous wastes. These "TSD" facilities, as they are called, are responsible for a great deal of documentation under RCRA. Therefore, Part II is essential for those Ashland locations designated as TSD facilities.

Slide 9

PART I

GENERATORS

The life of a hazardous chemical begins with the generator. All Ashland facilities, except those in the Carbon Black Division, are registered as generators.

Slide 10

It is the generator that identifies the waste as hazardous. All IC&S locations may use the lists found in SPI K-22 to identify some of the more commonly found hazardous wastes. Other divisions may find these lists in their Freight Classifications and Hazardous Designations manual, usually called the Freight Classifications Manual.

Slide 11

If the waste is not listed, another method of identifying it as hazardous is by comparing the characteristics of the waste to those criteria already given in the regulations. These criteria are ignitability, corrosivity, reactivity, and toxicity.

Slide 12 This can be determined by testing or by knowledge of the composition of the waste. For example, a waste mixture of flammable organic solvents would be classified as ignitable.

Slide 13 Prior to shipment, any drum containing a hazardous waste must display this yellow hazardous waste label.

Slide 14 Once the waste has been properly identified and labeled by the generator, a manifest can be prepared.

Slide 15 The manifest is the one link that binds the generator, the transporter, and the TSD facility together. It becomes the "birth certificate" of the hazardous waste. It shows what the waste is, where it comes from, its weight, container size, and other important information.

Slide 16 All generators are assigned a Hazardous Waste Identification Number by the US-EPA or by his state agency acting on the federal government's behalf. Generators must know the I.D. numbers of all participants in order to fill out the manifest properly. Specific details on filling out the manifest may be found in the SPI manual or the Freight Classifications manual for your division.

Slide 17 Even though the waste leaves the generator's location, the generator must still make sure that it will be disposed of properly.

To do this, he must make certain that the disposal or TSD facility has returned the original signed copy of the manifest showing exactly what has happened to the waste. If the generator does not receive this original within 35 days of shipment, he must trace the waste to determine its status.

Slide 18

If the waste cannot be located, the generator has 10 days to file an Exception Report with the EPA Regional Administrator, unless the original manifest is received as a result of the investigation.

Slide 19

All Ashland facilities registered as generators that ship hazardous waste off-site may have to submit an annual report to the EPA by March 1 for the preceding calendar year. Your plant operating records and completed manifest forms will give you the information needed to fill out this report. Check your SPI manual or Freight Classifications manual for details in filling out this report.

Slide 20

The IC&S Division is to keep all chemical waste disposal records permanently. This includes copies of signed manifests, annual reports, exception reports, test results, waste analyses, etc. RCRA requires these documents be kept at least three years.

Slide 21

Any facility that generates less than 1,000 kilograms (2,200 pounds) of hazardous waste per month is a small-quantity generator. This quantity includes customers' spent products that are stored over 10 days which will be recycled or disposed of and must be included in your records.

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Slide 22

This limited amount of hazardous waste need only be documented by written, dated waste production records or dated hazardous waste labels. A small-quantity generator is exempt from most RCRA regulations, but must still treat or dispose of the waste in a TSD facility according to RCRA or state standards. Once a small-quantity generator accumulates 1,000 kilograms of hazardous waste over any period of time, he is subject to most of the RCRA requirements.

Slide 23

TRANSPORTERS

The generator enlists the services of a transporter to move the hazardous waste from the generator's site to a treatment, storage, or disposal facility.

Slide 24

When transporting the waste – whether by air, rail, highway, or water– the manifest travels with the waste. The generator signs the manifest and keeps one copy, and the original and all other copies are given to the transporter.

Slide 25

Ashland facilities registered as transporters are generally involved with shipping their own wastes on Ashland trucks or with handling products returned by customers because of contamination. Your plant's "Notification of Hazardous Waste Activity" will tell you if you are registered as a transporter. If so, check with your state EPA to see if you should also register the trucks you use to haul hazardous waste.

Slide 26

Some plants will be accepting customer wastes for recovery or disposal. Although RCRA has established an exemption for wastes which are used, re-used, recycled, or reclaimed, this exemption does not apply to the RCRA listed wastes. These listed wastes are given in the SPI manual or the Freight Classifications manual in Subpart D, "Lists of Hazardous Wastes". Because these listed wastes are not exempt, many customers' wastes are still subject to RCRA requirements.

Slide 27

When your location uses an Ashland truck to transport recoverable or waste materials from a customer's plant, be sure to follow any D.O.T., state, and local requirements that apply to the material you are transporting. Any drums and containers used for shipping waste should be free of all Ashland labels, logos, or identification. These containers must also comply with proper gauge requirements and all D.O.T. requirements for packaging, marking, and labeling. Prior to shipment, all drums must display a hazardous waste label.

Slide 28

Don't accept any hazardous waste from your customer unless a manifest has been correctly completed. If your customer is a small-quantity generator, no manifest is necessary. Your plant can supply the customer with a blank manifest and help fill it out. If the customer has no EPA I.D. number, call Environmental Engineering in Dublin, Ohio for assistance. Remember to sign and date the manifest before leaving your customer's property.

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Slide 29

As the first transporter, your obligation is to take the waste to the second transporter, or to the TSD facility or alternate TSD facility stated on the manifest by the generator. If for some reason you can't carry out the original instructions on the manifest, contact your customer and revise the manifest according to his alternate instructions. If you're delivering the waste to a second transporter, keep a copy of the manifest for your location and give the original and remaining copies to the designated facility.

Slide 30

Once you've delivered the waste to either a second transporter or TSD facility, get the proper signature and the date of the delivery on the manifest. Your state regulations may require a completed copy of the manifest for their records also.

Slide 31

If there is a spill of the hazardous waste you're transporting, contain the spill immediately and notify local authorities. As the transporter, you are also responsible for cleaning up any hazardous waste discharges that occur during transportation and taking whatever action is required by Federal, state, or local authorities.

Slide 32

When the spill is of a type listed in SPI N-7, Report of Hazardous Material Incidents, contact the National Response Center. Their telephone number may be found in your Spill Prevention Control and

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Countermeasure Plan. A written report may also be required. For help in completing this report, call Fleet Services in Dublin, Ohio.

Slide 33 Now that we've discussed the responsibilities of generators and transporters, let's review each one separately.

Slide 34 The life of a hazardous waste begins at the generator's facility. The generator must:

Slide 35
- Identify and classify the hazardous waste.

Slide 36
- Label the waste as hazardous.

Slide 37
- Make sure that vehicles used to transport the waste are properly placarded.

Slide 38
- Fill out the manifest completely and accurately.

Slide 39
- Keep one copy of the manifest.

Slide 40
- Give the original and all extra copies of the manifest to the transporter.

Slide 41
- Submit an Exception Report to the E.P.A. if the original manifest is not returned or traced within 45 days.

Slide 42
- File an annual report with the E.P.A. by March 1 of each year for the preceding year if requested by the E.P.A.

Slide 43
- Retain all waste records for at least three years. IC&S locations retain waste records permanently.

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Slide 44 The transporter receives hazardous waste from the generator. As an Ashland transporter you should:

Slide 45 • Obtain the signature of the generator on the manifest.

Slide 46 • Keep the original and all extra copies of the manifest, giving the generator one copy.

Slide 47 • Follow all D.O.T., state, and local requirements when transporting hazardous waste materials.

Slide 48 • Remove all Ashland identification from containers used to hold waste material.

Slide 49 • Make certain that the hazardous waste label is displayed on the container prior to shipment.

Slide 50 • Refuse any hazardous waste from a customer who does not have a correctly completed manifest. Give assistance in completing the manifest if necessary.

Slide 51 • Once delivered, obtain the proper signature and date of delivery on the manifest.

Slide 52 • Transfer the original and all extra copies of the manifest to the next party, keeping one copy for your files.

Slide 53 By observing RCRA procedures, you will help to ensure the health and safety of your plant's employees, your customers, and all those who come in contact with hazardous waste handled by Ashland.

Slide 54 (Disclaimer)

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RCRA

Resource Conservation and Recovery Act

PART II

Treatment Storage and Disposal Facilities

July 1982

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Slide 1 (Ashland Logo)

Slide 2

THE RESOURCE CONSERVATION

AND RECOVERY ACT

PART II

Slide 3

As we continue with our program on RCRA regulations, we will be discussing those Ashland facilities that treat, store, or dispose of hazardous wastes.

Slide 4

Since this program is designed to give a basic overview of responsibilities placed on TSD facilities, we encourage you to refer to either your SPI manual or your Hazardous Designations and Freight Classifications manual, usually referred to simply as the Freight Classifications manual.

Slide 5

Following this presentation, a test will be given to assure your understanding of the information covered. Your test results will then become a permanent part of your personnel records.

Slide 6

TREATMENT, STORAGE,

AND DISPOSAL FACILITIES

Slide 7

All TSD facilities require a RCRA permit. As a treatment facility, you are involved with changing a hazardous waste physically, chemically, or biologically to make it less hazardous or non-hazardous.

Slide 8

The main treatment used by registered Ashland plants is the neutralization of waste.

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Slide 9

At this time RCRA does exempt certain types of neutralization processes from the permitting requirements. The neutralization of hazardous wastes which are listed only as being corrosive or as having corrosive characteristics are included in this exemption. Ashland plants are advised to follow the proposed security, inspection and general operating requirements for elementary neutralization.

Slide 10

Also exempt are waste water treatment tanks that receive, store, and treat waste waters that are hazardous waste or that generate, store, or treat a wastewater treatment sludge which is a hazardous waste. Permits are not needed in either of these special cases.

Slide 11

As a TSD facility you could be involved with storing hazardous waste temporarily until it is treated, disposed of, or stored somewhere else. If you store hazardous waste for over 10 days that is not generated by your plant, such as a customer's waste, a storage permit is needed.

Slide 12

You also need a permit if you store hazardous waste generated by your own plant for more than 90 days.

Slide 13

No permit is needed for storing your plant's waste less than 90 days, but you must document this storage with operating records for tanks and dated hazardous waste labels for drums. Most Ashland plants are not involved with the on-site disposal of hazardous waste.

Slide 14

If you are unsure whether your plant is a TSD facility under RCRA regulations, check to see if a Hazardous Waste Permit Application has been filed with the US-EPA for your location.

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Slide 15

All Notification of Hazardous Waste Activity forms for existing Ashland plants were sent to the US-EPA by Environmental Engineering, as were Hazardous Waste Permit applications for TSD plants. If a permit application was submitted for your plant, you currently have interim status until the permits are issued by the EPA.

Slide 16

On page one of Form 3 of your plant's copy of the permit application is listed whether your location is a treatment facility, indicated by a "T", or a storage facility, indicated by an "S". Check Section IIIB, Column A of the Processes Section, for this notation.

Slide 17

If your plant is a TSD facility, you must sign and date all manifests certifying acceptance of the hazardous waste described on the manifest. Note any discrepancies on the manifest.

Slide 18

Give the transporter his copy of the manifest, and keep your copy for your records.

Slide 19

Return the original manifest to the generator within 35 days after shipment.

Slide 20

If you accept a waste without a manifest, file an unmanifested waste report.

Slide 21

As a TSD facility in Interim Status, you must follow certain standards until your permit is issued. These standards are broken down into eight segments and affect most Ashland plants. They are: 1) General Plant Standards; 2) Preparedness and Prevention; 3) Contingency Plan and Emergency Procedures; 4) Manifest System, Recordkeeping, and Reporting; 5) Closure

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and Post-Closure; 6) Financial Requirements; 7) Use and Management of Containers; and 8) Tanks. We will now discuss what each of these Interim Standards means to you as a TSD facility.

Slide 22

(1. General Plant Standards)

Slide 23

Before you treat or store any hazardous waste, a detailed chemical and physical analysis of the waste must be run unless it can be classified based upon knowledge of the waste's components. You should repeat this analysis if the process generating the waste changes or when waste received does not match the manifest description.

Slide 24

A written waste analysis plan must be prepared that outlines the areas given in RCRA SPI K-22 or your Freight Classifications manual.

Slide 25

Because of the potential danger to people or livestock should any stored hazardous waste be discharged, TSD facilities must ensure the security of the storage and treatment areas. This includes a 24-hour surveillance system that continuously monitors and controls entry to these areas; or an artificial or natural barrier surrounding the area (e.g. a fence).

Slide 26

A means to control entry to the area must be present at all times. All entrances to these areas should have signs posted stating "DANGER - UNAUTHORIZED PERSONNEL KEEP OUT". If the entire plant is fenced, this sign should be placed at the entrance to the facility. There must be enough signs to be seen from any approach to the plant.

Slide 27

They must also be printed in English and any other language predominant in the surrounding geographical area, with print large enough to be seen from at least 25 feet.

Slide 28

Frequent inspections of your plant are necessary to make certain no wastes are being released into the environment. These inspections should be conducted often enough to identify malfunctions before health or the environment is harmed.

Slide 29

Develop a written schedule for inspecting all monitoring equipment, safety and emergency equipment, security devices, and operating and structural equipment that are used to prevent, detect, or respond to environmental or health hazards. Your schedule should be kept at the plant. It must identify types of problems which are to be looked for during the inspection.

Slide 30

The frequency of inspection for each item may vary, but should be based on the rate of possible deterioration of the equipment and the probability of an environmental or health incident if the malfunction goes undetected between inspections. Areas subject to spills, such as loading and unloading areas, must be inspected daily when in use.

Slide 31

Your inspection must at least include the items and frequencies given in Section 7, Use and Management of Containers, and Section 8, Tanks. If your inspection reveals any deterioration or malfunction of equipment or structures, remedy

the situation. When a hazard is imminent or has already occurred, correct the situation IMMEDIATELY.

Slide 32

As you perform your plant inspection, record the date and time, the name of the inspector, observations made, and the date and nature of any repairs or remedial actions. These inspection logs must be retained for the same length of time as other RCRA data.

Slide 33

Training personnel involved in waste treatment or storage is an important requirement under RCRA. This includes a complete program of classroom instruction or on-the-job training that teaches compliance with RCRA Interim Status Standards. This audiovisual program is designed to meet RCRA requirements.

As stated at the outset of this program, the test which accompanies Parts I and II will be used to verify each individual's understanding of the regulations. Any plant personnel involved in waste treatment or storage must complete this training program.

Slide 34

Once training is initially completed, the program is to be reviewed annually. Documentation of the training each employee receives must be kept at your location. The exact information needed is listed in SPI K-22 and in the Freight Classifications manual.

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Slide 35

One final topic to be considered under General Plant Standards is that of general requirements for ignitable, reactive, or incompatible wastes. Because of their flammable nature, these wastes must be kept away from sources of ignition or reaction. While ignitable or reactive waste is being handled, smoking and other sources of ignition must be kept away from designated "NO SMOKING" areas. These areas are to be clearly marked.

Slide 36

When mixing incompatible wastes and other materials, care must be taken not to generate extreme heat or pressure, fire or explosion, or any other violent reaction.

You must also be very cautious not to produce toxic mists, fumes, dusts, or gases sufficient enough to threaten the health of any individual within the area.

Other areas of risk to be avoided when mixing incompatible wastes are: damaging the waste container, producing uncontrolled flammable fumes or gases that might explode or catch fire, and any other reaction that could threaten health or the environment.

Slide 37

(2. Preparedness and Prevention)

Slide 38

The best way to approach the problems caused by hazardous wastes is to maintain and operate your treatment or storage facility safely. By minimizing the possibility of a fire, explosion,

or any unplanned release of hazardous waste, the chances of having an accident are greatly reduced. A list of equipment required under RCRA for TSD facilities may be found in your SPI or Freight Classifications manual.

Slide 39

Just having this equipment is not enough. It must also be tested and maintained in good working condition to assure proper operation in case of an emergency. Adequate aisle space is also needed to allow unobstructed movement of people and emergency equipment to any area of the plant.

Slide 40

Local police, fire departments, and emergency response teams should be made aware of your plant's layout; the types of waste handled and the hazards associated with each; the personnel work areas; and entrances to the facility and all evacuation routes.

Slide 41

If more than one local authority might respond to an emergency at your plant, designate a primary emergency authority with agreements by the other sources to provide support.

Slide 42

Local hospitals should also be familiarized with the kinds of waste your plant handles and the types of injuries or illnesses which could result from fires, explosions, or releases at your location. This can be done by sending these authorities copies of the RCRA Contingency Plan prepared by Environmental Engineering.

Slide 43

You should also obtain agreements with state emergency response teams, contractors, and equipment suppliers for their

support if needed. If any state or local authorities will not enter into this kind of agreement with your location, document their refusal.

Slide 44 (3. Contingency Plan and Emergency Procedures)

Slide 45 Your plant's current SPCC Plan will be amended with the assistance of Environmental Engineering to include all RCRA contingency and emergency procedures. Copies of this Contingency Plan must be sent to state and local authorities to provide them with all pertinent information regarding your plant's compliance with RCRA regulations.

Slide 46 (4. Manifest System, Recordkeeping, and Reporting)

Slide 47 Part I of this program outlines the manifest system. Your SPI or Freight Classifications manual contains complete details on procedures regarding manifest discrepancies, records to be kept, and reports that must be filed.

Slide 48 Recent "reduction of paperwork" regulations have proposed that a statistical sampling of TSD facilities file an annual report. Some states may also require their own annual or quarterly reports separate from this federal report. You will be contacted directly by state or federal authorities if you are to file an annual report. All records needed for this report should be kept in case your facility is one of the few chosen. For complete information on this procedure, check SPI K-22 or your Freight Classifications manual.

Slide 49 (5. Closure and Post-Closure)

Slide 50 A written closure plan outlining the steps necessary to

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completely close your plant must be kept at your plant location. Environmental Engineering is developing closure plans for TSD facilities.

Slide 51

Your location's closure plan is to be submitted to the EPA Regional Administrator at least 180 days prior to the beginning of plant closure.

Slide 52

In the event your plant is scheduled to close, there are several detailed instructions that must be followed under RCRA regulations. For this reason, contact Environmental Engineering at least seven months prior to the closure of your plant for assistance.

Slide 53

(6. Financial Requirements)

Slide 54

Each treatment or storage facility must have a written estimate of the cost of closing the plant in accordance with its closure plan. This estimate must be updated annually to reflect inflationary adjustments and changes in the closure plan itself. The federal government or individual states will require bonds or other means of financial assurance to provide the funds necessary for closure.

Slide 55

All TSD facilities must demonstrate financial responsibility for claims of personal injury or property damage resulting from accidents related to hazardous waste treatment or storage. This liability insurance is being handled through Corporate headquarters. Any questions regarding coverage can be directed to Environmental Engineering.

Slide 56 (7. Use and Management of Containers)

Slide 57 This section refers specifically to the use and management of containers. RCRA defines a container as any portable device in which a material is stored, transported, treated, disposed of, or otherwise handled. The following regulations apply only to plants that store hazardous waste.

Slide 58
- Hazardous waste stored in containers that leak or are in poor condition must be transferred to containers in good condition.

Slide 59
- The container must be made of or lined with materials that are compatible with the hazardous waste.

Slide 60
- Containers of hazardous waste must be closed during storage unless you are adding or removing waste.

Slide 61
- Do not open, handle, or store containers in such a way that they will rupture or cause the container to leak.

Slide 62
- Storage areas for containers must be inspected weekly.

Slide 63
- Containers holding ignitable or reactive waste must be located at least 50 feet from the plant's property line.

Slide 64
- Procedures for handling ignitable, reactive, or incompatible wastes, as outlined in your SPI or Freight Classifications manual, must be met.

Slide 65 (8. Tanks)

Slide 66 The following requirements apply to plants that use tanks to treat or store hazardous wastes. A concrete neutralization pit is

considered a tank under RCRA and must meet any of these regulations that apply.

Slide 67

- Don't place hazardous wastes or reagents in a tank if they could cause the tank or its liner to rupture, leak, or corrode.

Slide 68

- A tank must have a means to stop a continuous inflow, such as a waste feed cut-off system, a by-pass system, or a standby tank.

Slide 69

- Before treating or storing a substantially different waste, or before using a different chemical process for treatment or storage, you must conduct a waste analysis and trial treatment or storage test. Information from documented sources on similar storage or treatment processes may be substituted to show the proposed treatment or storage is compatible.

Slide 70

- Inspect discharge control equipment and data gathered from monitoring equipment at least once each operating day.

Slide 71

- Inspect the construction materials of the tank at least weekly for signs of corrosion or leakage. Also check the dike and surrounding area for signs of erosion or leakage.

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Slide 72

- If any deterioration or malfunction is found during inspection, remedy the problem immediately.

Slide 73

- Storage or treatment of ignitable or reactive waste in covered tanks must comply with the National Fire Protection Association's buffer zone requirements for tanks. Call Environmental Engineering for assistance on this.

Slide 74

- Don't place ignitable or reactive waste in a tank unless it is treated or mixed before or immediately after placing in the tank; or

Slide 75

Store ignitable or reactive wastes so that they are protected from any materials or conditions which may cause them to ignite or react.

Slide 76

- Don't place hazardous wastes in an unwashed tank which previously held an incompatible waste.

Slide 77

Requirements for uncovered tanks are given in your SPI or Freight Classifications manual.

Slide 78

When you receive your permit as a hazardous waste TSD facility, continue following the Interim Status Standards. As more standards are required, the Training Department, in cooperation with Environmental Engineering, will revise this program to reflect all changes.

Slide 79

Compliance with RCRA regulations will not only avoid severe criminal penalties under the law, but will also ensure a safe environment for all.

Slide 80

At this time, you may review any portions of this part of the program you do not fully understand. The test covering Part II will then be administered and added to your personnel file.

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ATTACHMENT II

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Research Manager Training Program

I. Training Conducted By

Ashland Chemical R&D Personnel
Ashland Chemical Company Environmental Engineering and/or Safety Services personnel, Columbus, OH
State Regulatory Representatives
Outside Seminars and Programs

II. Annual training at a seminar or meeting conducted by one or more of the above. Individual has responsibility for entire RCRA program at R&D Lab.

III. Corporate Memos

Memos from R&D Division, Environmental Engineering and Safety Services personnel are issued to keep managers up-to-date on current regulatory developments (e.g. OSHA, RCRA, etc.)

Contingency Plan from Environmental Engineering which outlines procedures to follow in hazardous waste emergencies

IV. Research Manager supervises monthly safety meeting.

V. Records of training

Maintained at the lab

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Research Services Coordinator Training Program

I. Training conducted by

Ashland Chemical R&D personnel

Ashland Chemical Company Environmental Engineering and/or Safety Services personnel, Columbus, OH

State regulatory representatives

Outside seminars and programs

II. Annual training at a seminar or meeting conducted by one or more of the above. Individual is responsible for implementation of the RCRA program.

III. Audio visual RCRA program (viewed at least annually)

Program reviewed and updated by Environmental Engineering, Columbus, OH

Emphasis on RCRA paperwork

IV. Corporate Memos

Review and understand memos from R&D Lab, Environmental Engineering and Safety Services Personnel

V. Monthly Safety Meeting

Supervise and conduct monthly safety meetings

VI. Records of Training

Maintained at the Lab

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Shipping-Receiving Clerk Training Program

I. Training conducted by

Research Services Coordinator

II. Annual Training

On-the-job by Research Services Coordinator

Review of RCRA A/V program

III. On-The-Job Training

*Review of manifest paperwork procedures by Research Services Coordinator. Individual is responsible for assisting with RCRA paperwork

IV. Audio Visual RCRA Program (Viewed at least annually)

Program reviewed and updated by Environmental Engineering, Columbus, OH

Emphasis on RCRA paperwork

V. Corporate Memos

Review and understand memos from R&D Division, Environmental Engineering, Safety Services personnel

Direction from Research Services Coordinator

R&D Safety Mannual

VI. Records

Files of current training maintained in the Lab

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Chemical Packaging Clerk Training Program

I. Training conducted by

Reserach Services Coordinator

II. Annual Training

On-The-Job by Research Services Coordinator

Review of RCRA A/V program

III. On-The-Job Training

Review of RCRA packaging procedures by Research Services Coordinator. Individual may assist in packaging wastes to DOT specifications

IV. Audio Visual RCRA Program (Viewed at least annually)

Program reviewed and updated by Environmental Engineering, Columbus, OH

Emphasis on RCRA paperwork

V. Corporate Memos

Review and understand memos from R&D Division, Environmental Engineering, Safety Services personnel

Direction from Research Services Coordinator

R&D Safety Manual

VI. Records

Files of current training maintained in the Lab

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Pilot Plant Group Leader Training Program

I. Training Conducted By

Research Services Coordinator

II. Annual Training on completion of Chemical Waste Profile Sheet. Individual is responsible for this aspect of RCRA program

III. Corporate Memos

Review and understand memos from R&D Division, Environmental Engineering and Safety Services personnel

R&D Safety Manual

IV. Monthly Safety Meeting

Conduct or supervise conduct of monthly safety meetings

V. Records of Training

Maintained at the Lab

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Senior Research Engineer Training Program

I. Training Conducted By

Pilot Plant Group Leader

II. Annual training on completion of Chemical Waste Profile Sheet. Individual is responsible for this area of RCRA program

III. Corporate Memos

Review and understand memos from R&D Division, Environmental Engineering and Safety Services personnel

R&D Safety Memos

IV. Monthly Safety Meeting

Conduct or supervise conduct of monthly safety meeting

V. Records of Training

Maintained at the Lab

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Research Engineer Training Program

I. Training Conducted By

Senior Research Engineer

II. Annual Training on completion of Chemical Waste Profile Sheet. Individual is responsible for this area of RCRA program

III. Corporate Memos

Review and understand memos from R&D Division, Environmental Engineering and Safety Services personnel

R&D Safety Manual

IV. Monthly Safety Meeting

Conduct or supervise conduct of monthly safety meetings

V. Records of Training

Maintained at the Lab

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Lab Technician

I. Training Conducted By

Research Engineer

II. Annual training on completion of Chemical Waste Profile Sheet. Individual is responsible for this area of RCRA program.

III. Corporate Information

R&D Safety Manual

IV. Monthly Safety Meeting

Conduct or supervise conduct of monthly safety meetings

V. Records of Training

Maintained at the Lab

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SECTION I

CLOSURE PLAN

AND

FINANCIAL REQUIREMENTS

FOR

ASHLAND CHEMICAL COMPANY

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HAZARDOUS WASTE STORAGE CLOSURE PLAN

The Resource Conservation And Recovery Act, RCRA, Regulations, Part 264 Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities and Part 265, Interim Status Standards for Owners and Operators of Hazardous Waste Treatment, Storage and Disposal Facilities, Subpart G requires preparation of a Closure and Post Closure Plan.

Ashland Chemical Company, Research Development Laboratory holding hazardous wastes longer than 90 days is classified and permitted as a Storage Facilities and is required to prepare a Closure and Post Closure Plan. The plan must cover the following topics as related to Storage Facilities:

1. Closure Performance Standard - Minimize need for further maintenance; control, minimize or eliminate continuing environmental concern (Paragraph 265.111)

2. Closure Plan - Identify how and when the facility will be closed or partially closed if applicable; satisfy stated closure requirements for containers, tanks. (Paragraph 265.112)

3. Notification - Intent to close; compliance with period of time prescribed for closure and other procedural requirements

4. Post Closure Plan - No requirements as long as all hazardous wastes are removed from the facility.

Ashland Chemical Company is engaged in storage of hazardous wastes in containers and a tank. Other than the standards prescribed for "Use and Management of Containers" Subpart I, (Paragraph 261.170-261.189) there are no specific requirements for closure of container storage facilities. Subpart J - Tanks, (Paragraphs 265.197) Closure requires removal of all hazardous waste and hazardous waste residues from tank, discharge control and confinement structures.

Subpart H Financial Requirements, (Paragraphs 264.142 and 265.142; and 264.143 and 265.143) require preparation of a written estimate of the cost of closure. This cost estimate must be available at the facility and must be updated annually using the government's published inflation factor. Financial Assurance for Facility Closure (Paragraphs 264.143 and 265.143) lists options by which an owner or operator can provide the necessary monies for closure as indicated by the cost estimate.

Liability Requirements (Paragraphs 264.147 and 265.147) mandates that an owner or operator of a hazardous waste storage facility must demonstrate financial responsibility for claims arising from sudden and accidental occurrences that cause injury to persons or people.

Evidence of Financial Assurance for Facility Closure and Liability Requirements must be submitted to the Regional Administrator by certified mail prior to July 6, 1982 and July 15, 1982, respectively.

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Location of and Description of Hazardous Waste Storage Facilities and Activities

The Ashland Chemical Company Dublin R&D Laboratory is located at 5200 Blazer Parkway, Dublin, Ohio 43017.

Container Storage Of Hazardous Wastes - The following wastes are stored in 55-gallon, DOT approved drums. The drums are inspected when placed in the storage area and on a weekly basis thereafter for evidence of leakage and/or container damage/deterioration:

D001	Ignitable wastes
D002	Corrosive wastes
F001	Spent halogenated solvents used in degreasing
F002	Spent halogenated solvents
F003	Spent non-halogenated solvents
F005	Spent non-halogenated solvents

A maximum of 400 55-gallon containers of hazardous waste could be in storage at any given time. Hazardous wastes will be stored an average of 180 days.

Bulk Storage of Hazardous Waste

Ignitable wastes (D001, F001, F002, F003, F005) are stored in an 8,000 gallon underground storage tank. The tank is used for storage of solvents used in pilot plant equipment cleaning and line flush. Accumulation within the tank is checked weekly. The material which is recovered from the tank is usually disposed of by incineration.

A maximum of 8,000 gallons of hazardous waste could be in the tank at any given time. The contents of this tank are stored over 90 days. The tank contents are gauged weekly.

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Closure Plan

Closure Performance Standard

The objective of this closure plan is to eliminate the need for post-closure care and prevent the escape of hazardous waste, hazardous waste constituents, leachate and contaminated rainfall to the ground and surface waters and the surrounding land.

All containerized hazardous wastes will be removed during closure. Any material in the container storage area containment system will be removed. If there is evidence of any spills or leaks outside of the containment systems, samples will be taken and analyzed to determine the extent of contamination in the soil and if necessary groundwater. Any contaminated soil will be removed and disposed of at a permitted disposal site.

This closure plan covers the closure of the waste container storage area. The maximum inventory of hazardous wastes which are expected at any one time are 400 drums of containerized wastes. This plan includes partial closure in the event that the waste container storage area is moved to another area of the plant.

This closure plan covers the closure of the underground waste solvent storage tank. The maximum inventory of hazardous wastes which are expected at any one time is 8,000 gallons in the solvent storage tank.

Closure Of Container Storage Area

All drums in the waste container storage area will be removed for transport to a permitted T/S/D facility. Five trips may be required to remove all waste drums.

Any liquids in the storage area containment system will be drummed, analyzed and depending on the analysis removed for disposal at a permitted T/S/D facility.

The drum storage area will be cleaned with a steam cleaner unit and an industrial grade detergent and water. We propose to wash the pad once with an industrial grade detergent and pick up all liquid, then flush the floor with clear water and vacuum it up. All liquid collected by the vacuum will be drummed off (we expect two 55-gallon drums to be generated) and the cleaner unit will be rinsed clean, also into the same drums. After all the scrubbing and rinsing is complete, we will test to show that the pad is clean in the following manner. A blank will first be run by saturating a clean sponge with distilled water. Water from the sponge will be analyzed for TOC and TOX. The sponge will then be resaturated and the previously cleaned pad will be manually scrubbed in several areas. The water on the pad will be picked up by the sponge, squeezed into a bottle, and then analyzed for TOC and TOX. If a major increase in analysis is noted between the blank and pad scrubbings, the pad will again be power scrubbed until tests show only minor differences. All of this work will be inspected by a P.E. and he will certify that the work has been done. All cleanup residues from the cleaning will be tested for pH and if they are below 2.0 or above 12.5 the aqueous solutions will be disposed of as a hazardous waste. If found hazardous, the liquid will be removed for disposal at an approved site.

Soils in the facility are not expected to be contaminated by container waste storage. However, if evidence of possible soil contamination exists, such as a break or hole in the pavement near the storage area or unloading area at least one soil sample will be taken in each area.

Closure of the Underground Waste Solvent Storage Tank

The maximum inventory of material in the underground tank at any one time is 8,000 gallons. Depending on the composition of the mixture it will be either sent to a reclaimer or sent to a disposal facility. The tank contents will be transferred to a tank wagon for transport.

The tank will be dug up, cleaned, dismantled and scrapped or reused within Ashland. The tank will be cleaned with a water rinse which will be circulated within the tank to clean all surfaces in the tank. The rinsate will be pumped into drums and disposed of as hazardous waste. All recovered material will be placed in containers that are compatible with the material and meet DOT requirements for shipping. If demolition of the tank is planned, the tank will be purged with air or nitrogen to insure that all volatile organic solvents are removed from the tank. This is necessary for personal protection of workers who may inhale the solvents and for removal of volatiles for the safe demolition of the tank.

Seven soil samples will be collected 0-12 inches below the tank. One sample will be at the center of the tank with 4 samples taken at each corner of the rectangular area of the tank. Two samples will be taken; outside the actual tank area at unpaved areas.

Soil samples will be collected using an auger; EPA Method 1.21.6. Soil samples will be analyzed using GC/MS Techniques for volatile organics; EPA Method 8240. It is not expected that groundwater will be encountered since the tank is not completely under the natural ground level. However, if groundwater is reached three samples of groundwater will be collected in lieu of the soil samples. Groundwater samples will be collected using a weighted bottle; EPA Method 1.2.1.2. Water samples will be analyzed using GC/MS Method 624.

Decontamination of Waste Tank

The waste storage tank will be cleaned with water as described in the previous section, Closure of the Underground Solvent Recovery Tank. All the rinsate will be collected and disposed of as hazardous waste at a permitted disposal site. Approximately 200 gallons of rinsate are expected from the decontamination process.

Following rinsing and air drying the tank and fittings will be dismantled. The empty tank and fittings will be disposed of as scrap or reused at this or another Ashland location.

Closure Notification And Schedule Of Closure

A closure plan must be submitted to the EPA Regional Administrator and Director of Ohio EPA at least 180 days before the date the plant is expected to begin closure. The Regional Administrator will then modify, approve or disapprove the plan within 90 days of receipt. Ashland and the affected public will have the opportunity to submit written comments on the closure plan prior to the Regional Administrator's decision on the plan.

Within 90 days after generating the final amount of hazardous waste, Ashland must treat or remove all hazardous waste from the site as described in the approved Closure Plan. Within six months after or generating the final hazardous waste, Ashland must complete the closure activities outlined in the Closure Plan. A longer closure period may be approved by the Regional Administrator or Ohio EPA Director.

Certification of closure must be submitted to the US/EPA Regional Administrator and the Director of the Ohio EPA when closure is completed. This certification must be completed both by Ashland and by an independent registered professional engineer that the facility (or hazardous waste activity at the plant) has been closed in accordance with the specifications in the approved Closure Plan.

Table One shows a schedule for closure even though we cannot predict the actual date of closure. All financial calculations are based on a closure date in the year 2002.

Final closure activities will be initiated within 90 days after generation of the final volume of hazardous wastes. The completion of closure will be within 180 days of this occurrence. The Ohio EPA will be notified 180 days before beginning final closure. Final closure will be supervised and certified by a professional engineer in addition to Ashland.

Post-Closure Plans

Post closure plans will not be needed for this facility because it is not a disposal facility.

Notice In Deed And Notice To Local Land Authority

Because this is only a hazardous waste storage facility and not a disposal facility notation is not necessary in the deed informing potential purchasers of restrictions associated with a disposal site.

Closure Cost Estimate

The closure cost estimates are presented in Table Two. Closure cost activities include removal of waste inventory, decontamination and cleanup of the waste container storage area, decontamination of the waste tank, the dismantling or removal of the waste tank, disposal of wash residue and closure certification. A 15% contingency has been added for the analysis, removal and disposal of any contaminated soil. A 15% contingency has also been added for administration overhead.

An estimated $123,285 (February, 1986 cost estimate) will be needed to close the hazardous waste storage facility.

The assumptions made in the cost estimate are as follows:

1.0 Removal of Waste Inventory - Drums

The waste volume is 400 drums of assorted wastes. Disposal costs are based on land disposal of the drums.

2.0 Hauling of Drummed Waste

Five trips will be required to remove the waste inventory.

3.0 Closure of the Waste Tank

The liquid volume is 8,000 gallon which will cost $.40/gallon for disposal. The cost of hauling to the disposal site is $.10/gallon. Approximately 8 hours at $100/hr will be required to dig up the tank.

4.0 Decontamination of Drum Storage Area

Any material in the storage area containment system will be removed. The container storage area will be decontaminated with a hot detergent wash. It is estimated that 32 manhours of labor will be required to clean up the storage area. Approximately six drums of wash residues will be accumulated. A disposal cost of $206 per drum is assumed for the wash residues which includes transportation.

5.0 Decontamination of Tank

The tank will be water cleaned. Approximately 200 gallons of residue and rinsate will be hauled away and disposed of for $3.73/gallon. Approximately 20 hours will be required to dismantle the tank. The dismantled tank will be disposed of as scrap or reused at this or another Ashland facility.

6.0 Closure Certification

The cost of certification of closure by a professional engineer is estimated on the basis of a labor rate of $60/hour and expenses for a 6-day period.

7.0 Contingencies

No soil contamination is anticipated as a result of waste container or tank storage and handling. Nevertheless, a 15% contingency has been added for the analysis, removal and disposal of contaminated soil. An additional 15% contingency has been added for administration overhead.

This closure cost estimate will be kept on file at this facility. It will be adjusted annually for inflation by using the Department Of Commerce's Annual Implicit Price Deflator for Gross National Product. The closure cost estimate will be adjusted for inflation by May 19 of each year and these numbers used to demonstrate financial responsibility which is due to be updated December 31 of each year. It will be revised whenever a change in the closure plan affects the cost of closure.

TABLE ONE

ANTICIPATED CLOSURE SCHEDULE

	Activity	Days																	
		10	20	30	40	50	60	70	80	90	100	110	120	130	140	150	160	170	180
1.0	Closure of Waste Tank																		
1.1	Removal of Final Waste Inventory																		
1.2	Decontaminate Tank																		
1.3	Removal of Clean-up Residues																		
1.4	Dismantle Tank																		
1.5	P.E. Certification of Closure																		
1.6	Certification of Closure and Submittal to U.S. EPA and Ohio EPA																		

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TABLE ONE

ANTICIPATED CLOSURE SCHEDULE

	Activity	Days																	
		10	20	30	40	50	60	70	80	90	100	110	120	130	140	150	160	170	180
2.0	Closure of Container Storage Area																		
2.1	Receipt of Final Volume of Hazardous Waste																		
2.2	Removal & Disposal of Final Waste Inventory																		
2.3	Decontamination & Clean-up of Container Storage Clean-up																		
2.4	Removal & Disposal of Wash Residue From Container Storage Clean-up																		
2.5	Soil Sampling																		
2.6	Removal of Contaminated Soil																		
2.7	Certification of Closure & Submittal to U.S. EPA and Ohio EPA																		

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TABLE TWO

ASHLAND CHEMICAL COMPANY

INDUSTRIAL CHEMICALS AND SOLVENTS DIVISION

COST ESTIMATE FOR FACILITY CLOSURE

February, 1986

1.0	Removal Of Final Waste Inventory (400 Drums Of Various Chemicals @ $200/Drum)	$80,000
2.0	Hauling (5 Trips @ $420/Trip)	2,100
3.0	Waste Tank	
	1.1 Removal of Final Waste Inventory (8,000 Gallons @ $.40/Gal)	3,200
	1.2 Hauling (8,000 Gallons @ $.10/Gal)	800
	1.3 Dig Up Tank (8 Hours @ $100/Hour)	800
	1.4 Dismantle the 8,000 Gallon Tank (20 Hours @ $12/Hour)	240
	1.5 Haul Away as Scrap to Disposal Site or Reuse at Ashland Facility (8,000 Lb @ $.30/Lb)	2,400
	Sub Total	$ 7,440
4.0	Decontamination Of Drum Storage Area	
	Labor (32 Hours @ $12/Hour)	384
	Rental Of Steam Cleaner (2 Day @ $75/Day)	150
	Disposal Of Contamination Residues (2 Drums @ $206/Drum)	412
	Sub Total	$ 946
5.0	Decontamination of Tank	
	5.1 Labor (4 Hours @ $12/Hour)	48
	5.2 Rental of Steam Cleaner (1 Day @ $75/Day)	75
	5.3 Transportation & Disposal of Rinsate (200 Gallons @ $3.73 Gallon, Includes Transportation)	746
	Sub Total	$ 869

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TABLE TWO

ASHLAND CHEMICAL COMPANY

INDUSTRIAL CHEMICALS AND SOLVENTS DIVISION

COST ESTIMATE FOR FACILITY CLOSURE

February, 1986

(Continued)

6.0	Closure Certification	
	Labor (P.E. 48 Hours @ $60/Hour)	2,880
	Expenses (6 Days @ $75/Day)	450
	Transportation (600 Mile @ $0.25/Mile)	150
	Sub Total	$ 3,480
	Sub Total (1.0 - 6.0)	$94,835
7.0	Contingencies	
	15% Administration Overhead	14,225
	15% Analysis, Removal, & Disposal Of Contaminated Soil	14,225
8.0	Total Closure Cost	$123,285

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Financial Assurance Mechanism

Ashland uses the financial test and corporate guarantee to demonstrate financial assurance both for facility closure and liability requirements. The financial test and corporate guarantee is attached.

The financial test for December 31, 1985, which is 90 days after Ashland's fiscal year, will show this revised cost estimate.

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Region V FAM letter

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of Region V FAM letter

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Ashland

ASHLAND OIL, INC. • POST OFFICE BOX 391 • ASHLAND, KENTUCKY • 41114 • PHONE (606) 329-3333

WILLIAM R. SEATON
Vice Chairman of the Board
(606) 329-3733

December 19, 1985

Director
Ohio Environmental Protection Agency
361 E. Broad Street
Columbus, Ohio 43216-1049

Dear Director:

I am the chief financial officer of Ashland Oil, Inc. P.O. Box 391, Ashland, Kentucky 41114. This letter is in support of the use of the financial test to demonstrate financial responsibility for liability coverage and closure and/or post-closure care as specified in Chapters 3745-55 and 3745-66 of the Administrative Code.

The owner or operator identified above is the owner or operator of the following facilities for which liability coverage is being demonstrated through the financial test specified in Chapters 3745-55 and 3745-66 of the Administrative Code:

EPA Region	Plant Name & Address	EPA ID Number
V	Ashland Chemical Co IC&S 200 Darrow Road Akron, Ohio 43305	OHD000723973
	Ashland Chemical Co IC&S 2788 Glendale-Milford Rd Cincinnati, Ohio 45241	OHD000289912
	Ashland Chemical Co IC&S 2945 East 83rd Street Cleveland, Ohio 41104	OHD080166002
	Ashland Chemical Co IC&S 3849 Fisher Road Columbus, Ohio 43228	OHD000816736
	Ashland Chemical Co IC&S 2854 Springboro Pike Moraine, Ohio 45439	OHD074700311
	Ashland Chemical Co R&D Laboratory 5200 Blazer Parkway Dublin, Ohio 43017	OHD042311209

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EPA Region	Plant Name & Address	EPA ID Number
V	Ashland Chemical Company Specialty Polymers & Adhesives Div 1745 Cottage St. Ashland, Ohio 44805-1298	OHD071114375

1. The owner or operator identified above owns or operates the following facilities for which financial assurance for closure or post-closure care is demonstrated through the financial test psecified in Chapters 3745-55 or 3745-66 of the Administrative Code. The current closure and/or post-closure cost estimates covered by the test are shown for each facility:

EPA Region	Plant Name & Address	EPA ID Number	Closure Cost Estimate
V	Ashland Chemical Co IC&S 200 Darrow Road Akron, Ohio 43305	OHD000723973	$ 102,662
	Ashland Chemical Co IC&S 2788 Glendale-Milford Rd Cincinnati, Ohio 45241	OHD000289912	$ 13,696
	Ashland Chemical Co IC&S 2945 East 83rd Street Cleveland, Ohio 44104	OHD080166002	$ 51,000
	Ashland Chemical Co IC&S 3849 Fisher Road Columbus, Ohio 43228	OHD000816736	$ 24,628
	Ashland Chemical Co IC&S 2854 Springboro Pike Moraine, Ohio 45439	OHD074700311	$ 25,600
	Ashland Chemical Co R&D Laboratory 5200 Blazer Parkway Dublin, Ohio 43017	OHD042311209	$ 61,017
	Ashland Chemical Company Specialty Polymers & Adhesives Div 1745 Cottage St. Ashland, Ohio 44805-1298	OHD071114375	$ 1,964

2. The owner or operator identified above guarantees, through the corporate guarantee specified in Chapters 3745-55 and 3745-66 of the Administrative Code, the closure and post-closure care of the following facilities owned or operated by its subsidiaries. The current cost estimates for the closure or post-closure care so guaranteed are shown for each facility: None

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3. The owner or operator identified above owns or operates the following hazardous waste management facilities for which financial assurance for closure or, if a disposal facility, post-closure care, is not demonstrated the director through the financial test or any other financial assurance mechanism specified in Chapters 3745-55 or 3745-66 of the Administrative Code. The current closure and/or post-closure cost estimates not covered by such financial assurance are shown for each facility: None.

This owner or operator is required to file a Form 10K with the Securities and Exchange Commission (SEC) for the latest fiscal year.

The fiscal year of this owner or operator ends on September 30. The figures for the following items marked with an asterisk are derived from this owner's or operator's independently audited, year-end financial statements for the latest completed fiscal year, ended September 30, 1985.

Part B Closure or Post Closure Care and Liability Coverage

Alternative II

1.	Sum of Current closure and post-closure cost estimates. (Total of all cost estimates listed above.)	$ 5,485,514
2.	Amount of annual aggregate liability coverage to be demonstrated.	$ 8,000,000
3.	Sum of lines 1 and 2	$ 13,485,514
4.	Current bond rating of most recent issuance and name of rating service	A3 - Moody's
5.	Date of issuance of bond	August 1, 1982
6.	Date of maturity of bond	August 1, 2012
*7.	Tangible net worth (if any portion of the closure and post-closure cost estimates is included in "total liabilities" on your firm's financial statements, you may add the amount of that portion to this line)	$ 843,298,000
*8.	Total assets in U.S. (required only if less than 90% of firm's assets are located in the U.S.)	

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	Yes	No
9. Is line 7 at least $10 million?	X	
10. Is line 7 at least 6 times line 3?	X	
*11. Are at least 90% of firm's assets located in the U.S.? If not, complete line 12.	X	
12. Is line 8 at least 6 times line 3?		

I hereby certify that the wording of this letter is identical to the wording specified in paragraph (G) of Rule 3745-55-51 of the Administrative Code as such regulations were constituted on the date shown immediately below.

William R. Seaton
Chief Financial Officer and
Vice Chairman of the Board
December 19, 1985

RW

WRS:kl

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Ernst & Whinney

1900 Meidinger Tower
Louisville Galleria
Louisville, Kentucky 40202

502/583-0251

Ashland Oil, Inc.
Ashland, Kentucky

At your request, we have performed the procedures enumerated below with respect to selected financial data of Ashland Oil, Inc. and subsidiaries (Ashland) as of September 30, 1985, to assist you in reporting under various state and federal environmental protection standards and regulations. We made an examination of Ashland's consolidated financial statements in accordance with generally accepted auditing standards and, in our report dated November 6, 1985, expressed an unqualified opinion on Ashland's consolidated financial statements for the year ended September 30, 1985 from which the specified data was derived. The procedures were performed solely for the purpose stated above and this report is not to be referred to or used for any other purpose. The financial data, which is as of Ashland's September 30, 1985 year-end, subjected to our procedures is as follows (in thousands):

Total assets	$3,927,633
Less intangible assets	86,113
Tangible assets	3,841,520
Total liabilities and redeemable preferred stock	2,998,222
Tangible net worth	$ 843,298

The procedures we performed relative to the above figures are summarized as follows:

a. We compared the amount presented above as total assets to the amount for total assets in the September 30, 1985 audited financial statements noting them to be in agreement.

b. We summarized amounts for intangible assets from schedules furnished by Ashland and noted the total of such amounts agreed with the amount for total intangible assets set forth above.

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c. The individual amounts comprising the detail of intangible assets as set forth on the summary referred to in (b) above were compared to amounts included in the September 30, 1985 audited financial statements under various captions and found to be in agreement.

d. We added the individual amounts for current liabilities, noncurrent liabilities and deferred credits and redeemable preferred stock as set forth in the September 30, 1985 audited financial statements and compared the total of such amounts to the amount presented above for total liabilities and redeemable preferred stock and found them to be in agreement.

Additionally, schedules were furnished by Ashland summarizing data derived from their financial statements at September 30, 1985 concluding that at least ninety percent of Ashland's assets were located in the United States. Amounts on these schedules were compared to amounts included in the September 30, 1985 audited financial statements under various captions and found to be in agreement.

Because the above procedures do not constitute an examination in accordance with generally accepted auditing standards, we do not express an opinion on the tangible assets, tangible net worth or percent of assets located in the United States as stated above. In connection with the procedures referred to above, no matters came to our attention that caused us to believe that such amounts should be adjusted. Had we performed additional procedures or had we made an examination of the tangible assets, tangible net worth or percent of assets located in the United States in accordance with generally accepted auditing standards, other matters might have come to our attention that would have been reported to you. This report relates only to the accounts and items specified above and does not extend to the consolidated financial statements of Ashland, taken as a whole.

Ernst & Whinney

Louisville, Kentucky
November 6, 1985

CLOSURE COST ESTIMATES

FOR TSD FACILITIES BY EPA REGION

ASHLAND CHEMICAL PLANTS

EPA Region	Plant Name & Address	EPA ID Number	Closure Cost Estimate
II	Ashland Chemical Company IC&S 221 Foundry Street Newark, New Jersey 07105	NJD060803905	$ 11,708
	Ashland Chemical Company IC&S 3 Broad Street Binghamton, New York 13902	NYD049253719	$ 28,618
	Ashland Chemical Company IC&S 3701 River Road Tonawanda, New York 14150	NYD000688630	$ 11,708
III	Ashland Chemical Company Electronics & Laboratory Products Division Hellertown Road (Glendon) Easton, Pennsylvania 18042	PAD043394683	$ 13,158
	Ashland Chemical Company IC&S 150 West Fourth Avenue Freedom, Pennsylvania 15042	PAD000797548	$ 29,250
	Ashland Chemical Company IC&S 2410 Patterson Avenue S.W. Roanoke, Virginia 24016	VAD062373600	$ 35,200
IV	Ashland Chemical Company IC&S 3300 Ball Street Birmingham, Alabama 34234	ALD061149712	$ 28,805
	Ashland Chemical Company IC&S 701 Western Drive Mobile, Alabama 36607	ALD059661454	$ 22,926
	Ashland Chemical Company IC&S 1645 North McDuff Avenue Jacksonville, Florida 32205	FLD079859492	$ 15,114
	Ashland Chemical Company IC&S 5125 West Hanna Avenue Tampa, Florida 33614	FLD067230771	$ 12,270

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Region	Plant Name & Address	EPA ID Number	Closure Cost Estimate
	Ashland Chemical Company IC&S 4550 Northeast Expressway Doraville, Georgia 30340	GAD066465824	$ 25,940
	Ashland Chemical Company IC&S 400 Telfair Road Savannah, Georgia 31401	GAD041007063	$ 27,601
	Ashland Chemical Company IC&S 100 12th Street Catlettsburg, Kentucky 41129	KYD000828806	$ 11,676
	Ashland Chemical Company IC&S 4185 Algonquin Parkway Louisville, Kentucky 40211	KYD024041063	$ 12,535
	Ashland Chemical Company IC&S 455 Industrial Drive Jackson, Mississippi 39209	MSD000829150	$ 11,780
	Ashland Chemical Company IC&S 3930 Glenwood Drive Charlotte, North Carolina 28208	NCD061263315	$ 30,215
	Ashland Chemical Company IC&S 2802 Patterson Street Greensboro, North Carolina 27407	NCD024599011	$ 14,239
	Ashland Chemical Company IC&S 1415 South Bloodworth Street Raleigh, North Carolina 27601	NCD088560032	$ 26,978
	Ashland Chemical Company 729 Mauney Drive Columbia, South Carolina 29201	SCD900839823	$ 39,118
	Ashland Chemical Company IC&S 100 Industrial Boulevard Greenville, South Carolina 29607	SCD062697735	$ 25,151
	Ashland Chemical Company IC&S 5263 National Drive Knoxville, Tennessee 37914	TND095058541	$ 34,870
	Ashland Chemical Company IC&S 2351 Channel Avenue Memphis, Tennessee 38106	TND048319644	$ 41,270
V	Ashland Chemical Company Chemical Systems Division 142nd & Paxton Avenue Calumet City, Illinois 60409	ILD043369446	$ 16,033

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Region	Plant Name & Address	EPA ID Number	Closure Cost Estimate
	Ashland Chemical Company IC&S 8500 S. Willow Springs Road Willow Springs, Illinois 60480	ILD980700538	$ 32,012
	Ashland Chemical Company IC&S 2416 Pennsylvania Street Ft. Wayne, Indiana	IND000810705	$ 5,854
	Ashland Chemical Company IC&S 8315 East 33rd Street Indianapolis, Indiana 46226	IND040300964	$ 18,900
	Ashland Chemical Company IC&S 1817 West Indiana Avenue South Bend, Indiana 46613	IND016621476	$ 34,453
	Ashland Chemical Company IC&S 6451 Georgia Avenue Detroit, Michigan 48211	MID040573677	$ 30,070
	Ashland Chemical Company IC&S 1800 Glenrose Avenue Lansing, Michigan 48915	MID047173653	$ 375,000
	Ashland Chemical Company IC&S 4401 Valley Industrial Boulevard Shakopee, Minnesota 55379	MND000686709	$ 20,233
	Ashland Chemical Company IC&S 200 Darrow Road Akron, Ohio 44305	OHD000723973	$ 102,662
	Ashland Chemical Company Specialty Polymers & Adhesives 1745 Cottage St. Ashland, OH 44805-1298	OHD071114375	$ 1,964
	Ashland Chemical Company IC&S 2788 Glendale-Milford Road Cincinnati, Ohio 45241	OHD002899912	$ 13,696
	Ashland Chemical Company IC&S 2945 East 83rd Street Cleveland, Ohio 44104	OHD080166002	$ 51,000
	Ashland Chemical Company IC&S 3849 Fisher Road Columbus, Ohio 43228	OHD000816736	$ 24,628
	Ashland Chemical Company IC&S 2854 Springboro Pike Moraine, Ohio 45439	OHD074700311	$ 25,600

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Region	Plant Name & Address	EPA ID Number	Closure Cost Estimate
	Ashland Chemical Company R&D Laboratory 5200 Blazer Parkway Dublin, Ohio 43017	OHD042311209	$ 61,017
	Ashland Chemical Company IC&S 204 Madison Street Menasha, Wisconsin 54952	WID000713602	$ 18,300
	Ashland Chemical Company IC&S 1033 North Hawley Road Milwaukee, Wisconsin 53208	WID053689196	$ 30,500
VI	Ashland Chemical Company IC&S 4300 Southwest 36th Street Oklahoma City, Oklahoma 73119	OKD042146324	$ 12,879
	Ashland Chemical Company IC&S 3535 West 21st Street Tulsa, Oklahoma 74107	OKD041502089	$ 12,879
	Ashland Chemical Company IC&S 3101 Wood Drive Garland, Texas 75041	TXD980745095	$ 37,252
	Ashland Chemical Company IC&S 8901 Old Galveston Road Houston, Texas 70034	TXD095191920	$ 13,708
	Ashland Chemical Company IC&S Rankin Highway, Route 1 Midland, Texas 79701	TXD000761031	$ 11,708
VII	Ashland Chemical Company IC&S 5420 Speaker Road Kansas City, Kansas 66106	KSD057889313	$ 25,300
	Ashland Chemical Company IC&S 7710 Polk Street St. Louis, Missouri 63111	MOD031005341	$ 49,400
VIII	Ashland Chemical Company IC&S Freeport Center Building 12 Clearfield, Utah 84016	UTD048406144	$ 35,891
IX	Ashland Chemical Company Polyesters Division 6608 East 26th Street Los Angeles, California	CAD044046274	$ 5,854

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Region	Plant Name & Address	EPA ID Number	Closure Cost Estimate
	Ashland Chemical Company IC&S 8600 Enterprise Drive Newark, California 94560	CAD066562521	$ 14,858
	Ashland Chemical Company IC&S 10505 South Painter Avenue Santa Fe Springs, CA 90670	CAD044061422	$ 18,733
		TOTAL	$1,610,514

	PLANT NAME & ADDRESS	EPA ID NUMBER	CLOSURE COST EST
IV	Ashland Chemical Co IC&S 100 12th Street Catlettsburg, Ky 41129	KYD000828806	$11,676
	Ashland Chemical Co IC&S 4185 Algonquin Parkway Louisville, KY 40211	KYD024041063	$12,535
	Ashland Petroleum Company P.O. Box 391 Ashland, Ky 41114 Location: Route 3 Catlettsburg, KY	KYD000615898	Closure Cost $808,000 Post Closure Cost $149,000
	Viney Branch Catlettsburg, KY	KYD000615906	Closure Cost $2,918,000 Post Closure Cost - 0 -
		TOTAL	$5,485,514

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SECTION J

OTHER FEDERAL LAWS

	Law	Impact Upon The Permit
1.	Wild And Scenic Rivers Act	Not Applicable
2.	National Historic Preservation Act Of 1966	Not Applicable
3.	Endangered Species Act	Not Applicable
4.	Coastal Zone Management Act	Not Applicable
5.	Fish And Wildlife Coordination Act	Not Applicable

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CERTIFICATION

I hereby certify that I have reviewed this Part B permit application and being familiar with the provisions of 40 CFR Part 264 attest that this application has been prepared in accordance with good engineering practices. This application has been prepared by Arlene A. Hendrickson, Environmental Engineer. The review and evaluation is for the Ashland Chemical Company, R&D Laboratories located at 5200 Blazer Parkway, Dublin, OH.

ELDON RONNING
Print Name Of Registered Professional Engineer

Eldon E. Ronning
Signature Of Registered Professional Engineer

Registration No.: E-49075

State: Ohio

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CERTIFICATION

This is to certify that under penalty of law I have personally examined and am familiar with the information submitted in this document and all attachments and that, based on my inquiry of those individuals immediately responsible for obtaining the information, I believe 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.

The certification is for the Ashland Chemical Company, Research & Development Laboratry, 5200 Blazer Parkway, Dublin, OH. The application has been prepared by Arlene A. Hendrickson, Environmental Engineer.

Signature Of The Operator Or Authorized Representative

James D. Idol, Jr., Vice President, Venture R&D
Name And Title (Please Type Or Print)

Date: 11/20/84 Telephone No.: (614) 889-3188

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CERTIFICATION

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to be the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

The certification is for the Ashland Chemical Company, Research & Development Laboratory, 5200 Blazer Parkway, Dublin, OH. The application has been prepared by Arlene A. Hendrickson, Environmental Engineer.

Mr. James D. Idol Jr.
Signature Of The Operator Or Authorized Representative

James D. Idol, Jr., Vice President, Venture R&D
Name And Title (Please Type Or Print)

Date: 25 Feb 1986 Telephone No.: (614) 889-3188