2B

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REVISED PART A

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

LABEL ITEMS

I. EPA I.D. NUMBER

III. FACILITY NAME

V. FACILITY MAILING ADDRESS

VI. FACILITY LOCATION

PLEASE PLACE LABEL IN THIS SPACE

GENERAL INSTRUCTIONS

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

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	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		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	
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		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		
E. Does or will this facility treat, store, or dispose of hazardous wastes? (FORM 3)	X		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	
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		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	
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		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 PROTECCION TECNICA ECOLOGICA, INC.

IV. FACILITY CONTACT

A. NAME & TITLE *(last, first, & title)*	B. PHONE *(area code & no.)*
2 FERNANDEZ, JORGE PRESIDENT	809 836 2058

V. FACILITY MAILING ADDRESS

A. STREET OR P.O. BOX

3 FIRM DELIVERY

B. CITY OR TOWN	C. STATE	D. ZIP CODE
4 PONCE	PR	00731

VI. FACILITY LOCATION

A. STREET, ROUTE NO. OR OTHER SPECIFIC IDENTIFIER

5 ROAD 385 KM 35

B. COUNTY NAME

TALLABOA

C. CITY OR TOWN	D. STATE	E. ZIP CODE	F. COUNTY CODE *(if known)*
6 PENUELAS	PR	00724	

CONTINUE ON REVE

A. FIRST	B. SECOND
7 7399 (specify) Business Service (NEC) Waste Disposal	7 8899 (specify) Service (NEC) Landfill
C. THIRD	**D. FOURTH**
7 (specify)	7 (specify)

VIII. OPERATOR INFORMATION

A. NAME: 8 PROTECCION TECNICA ECOLOGICA, INC.

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

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

D. PHONE (area code & no.): A 809 837 2211

E. STREET OR P.O. BOX: ROAD 385 Km35

F. CITY OR TOWN: B PENEULAS

G. STATE: PR

H. ZIP CODE: 00731

IX. INDIAN LAND

Is the facility located on Indian lands? [ ] YES [X] NO

X. EXISTING ENVIRONMENTAL PERMITS

A. NPDES (Discharges to Surface Water): 9 N	**D. PSD** (Air Emissions from Proposed Sources): 9 P
B. UIC (Underground Injection of Fluids): 9 U	**E. OTHER** (specify): 9 R'S'P' # 5 (specify) PUERTO RICO ENVIRONMENTAL QUALITY BOARD
C. RCRA (Hazardous Wastes): 9 R	**E. OTHER** (specify): 9 75-12-19-56 (specify) Puerto Rico Planning Board

XI. MAP

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

XII. NATURE OF BUSINESS (provide a brief description)

Treatment, storage and disposal of hazardous and non-hazardous waste from various manufacturing plants and government in Puerto Rico.

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
Jorge J. Fernandez, President	Jorge J. Fernández Porto	4-1-86

COMMENTS FOR OFFICIAL USE ONLY

C

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 PRD091018622

FOR OFFICIAL USE ONLY

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

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 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 data)*

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

☐ 2. NEW 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)*

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

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		1

LINE NUMBER	A. PROCESS CODE (from list above)	B. PROCESS DESIGN CAPACITY 1. AMOUNT (specify)	B. PROCESS DESIGN CAPACITY 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. PROCESS DESIGN CAPACITY 2. UNIT OF MEASURE (enter code)	FOR OFFICIAL USE ONLY
X-1	S02	600	G		5	T04	216 CY/ DAY		
X-2	T03	20	E		6	D80	620	A	
1	T01	20,000	U		7	S04	289,500	G	
2	S01	84,480	G		8				
3	S02	164,000	G		9				
4	T02	10,000	U		10				

III. PROCESSES *continued*

C. SPACE FOR ADDITIONAL PROCESS CODES OR FOR DESCRIBING OTHER PROCESSES (CODE T04 FOR EACH PROCESS ENTERED HERE INCLUDE DESIGN CAPACITY

T 04 Stabilization/fixation process (216 CY/DAY Output Rating)

IV. DESCRIPTION OF HAZARDOUS WASTES

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

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

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

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

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

D. PROCESSES

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

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

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

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

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

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

CONTINUE ON PAGE 3

EPA I.D. NUMBER (enter from page 1): W P R D 0 9 1 0 1 8 6 2 2 — T/A/C 1

FOR OFFICIAL USE ONLY: W DUP — T/A/C 2 DUP

IV. 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	D001	230,000	K	S01 S02 T04 D80*	
2	D002	4,000,000	K	S01 S02 T04 D80*	
3	D004	100	K	S01 S02 T04 D80*	
4	D005	100	K	S01 S02 T04 D80*	
5	D006	300	K	S01 S02 T04 D80*	
6	D007	50	K	S01 S02 T04 D80*	
7	D008	950,000	K	S01 S02 T04 D80*	
8	D009	55,000	K	S01 S02 T04 D80*	
9	D010	200	K	S01 S02 T04 D80*	
10	D011	10	K	S01 S02 T04 D80*	
11	D012	100	K	S01 S02 T04 D80*	
12	D013	290,000	K	S01 S02 T04 D80*	
13	D014	100	K	S01 S02 T04 D80*	
14	D015	100	K	S01 S02 T04 D80*	
15	D016	100	K	S01 S02 T04 D80*	
16	D017	100	K	S01 S02 T04 D80*	
17	F001	64,000	K	S01 S02 T04 D80*	
18	F002	17,000	K	S01 S02 T04 D80*	
19	F003	60,000	K	S01 S02 T04 D80*	
20	F004	100	K	S01 S02 T04 D80*	
21	F005	31,000	K	S01 S02 T04 D80*	
22	F006	52,000	K	S01 S02 T04 D80*	
23	F019	100	K	S01 S02 T04 D80*	
24	F024	100	K	S01 S02 T04 D80*	
25	K061	100	K	S01 S02 T04 D80*	
26	K062	2,000	K	S01 S02 T04 D80*	

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*Additionally, Process Codes of T01, T02, S04

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

CONTINUE ON REVERSE

EPA I.D. NUMBER (enter from page 1): WPRD091018622 T/A 1

FOR OFFICIAL USE ONLY: W DUP T/A 2 DUP

IV. 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	K069	100	K	S01 S02 T04 D80*	
2	K100	100	K	S01 S02 T04 D80*	
3	K084	100	K	S01 S02 T04 D80*	
4	K101	100	K	S01 S02 T04 D80*	
5	K102	100	K	S01 S02 T04 D80*	
6	K086	100	K	S01 S02 T04 D80*	
7	K002	100	K	S01 S02 T04 D80*	
8	K003	100	K	S01 S02 T04 D80*	
9	K004	100	K	S01 S02 T04 D80*	
10	K005	100	K	S01 S02 T04 D80*	
11	K005	100	K	S01 S02 T04 D80*	
12	K006	100	K	S01 S02 T04 D80*	
13	K007	100	K	S01 S02 T04 D80*	
14	K008	100	K	S01 S02 T04 D80*	
15	K009	100	K	S01 S02 T04 D80*	
16	K010	100	K	S01 S02 T04 D80*	
17	K014	100	K	S01 S02 T04 D80*	
18	K015	100	K	S01 S02 T04 D80*	
19	K016	100	K	S01 S02 T04 D80*	
20	K017	100	K	S01 S02 T04 D80*	
21	K018	100	K	S01 S02 T04 D80*	
22	K019	100	K	S01 S02 T04 D80*	
23	K020	100	K	S01 S02 T04 D80*	
24	K021	100	K	S01 S02 T04 D80*	
25	K022	100	K	S01 S02 T04 D80*	
26	K023	100	K	S01 S02 T04 D80*	

EPA Form 3510-3 (6-80)

CONTINUE ON REVERSE

* Additionally , Process Cods of T01, T02, S04

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

NOTE Photocopy this page before completing if you have more than 26 wastes to list. Form Approved OMB No. 158-S80024

EPA I.D. NUMBER (enter from page 1)	FOR OFFICIAL USE ONLY
WPRD091018622 T/A C 1	W DUP T/A C 2 DUP

IV. 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	K024	100	K	S01 S02 T04 D80*	
2	K093	100	K	S01 S02 T04 D80*	
3	K094	100	K	S01 S02 T04 D80*	
4	K025	100	K	S01 S02 T04 D80*	
5	K026	100	K	S01 S02 T04 D80*	
6	K028	100	K	S01 S02 T04 D80*	
7	K029	100	K	S01 S02 T04 D80*	
8	K095	100	K	S01 S02 T04 D80*	
9	K096	100	K	S01 S02 T04 D80*	
10	K030	100	K	S01 S02 T04 D80*	
11	K083	100	K	S01 S02 T04 D80*	
12	K103	100	K	S01 S02 T04 D80*	
13	K085	100	K	S01 S02 T04 D80*	
14	K105	100	K	S01 S02 T04 D80*	
15	K071	100	K	S01 S02 T04 D80*	
16	K073	100	K	S01 S02 T04 D80*	
17	K106	100	K	S01 S02 T04 D80*	
18	K031	100	K	S01 S02 T04 D80*	
19	K032	100	K	S01 S02 T04 D80*	
20	K033	100	K	S01 S02 T04 D80*	
21	K034	100	K	S01 S02 T04 D80*	
22	K097	100	K	S01 S02 T04 D80*	
23	K048	100	K	S01 S02 T04 D80*	
24	K049	100	K	S01 S02 T04 D80*	
25	K050	100	K	S01 S02 T04 D80*	
26	K051	100	K	S01 S02 T04 D80*	

EPA Form 3510-3 (6-80) CONTINUE ON REVERSE

* Additionally, Process Codes of T01 T02, S04

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

NOTE: Photocopy this page before completing if you have more than 26 wastes to list. Form Approved OMB No. 158-80-004

EPA I.D. NUMBER (enter from page 1)	FOR OFFICIAL USE ONLY
WPRD091018622 2 1	W DUP 2 DUP

IV. 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	K052	2,000	K	S01 S02 T04 D80*	
2	U001	100	K	S01 S02 T04 D80*	
3	U002	5,000	K	S01 S02 T04 D80*	
4	U003	100	K	S01 S02 T04 D80*	
5	U004	100	K	S01 S02 T04 D80*	
6	U005	100	K	S01 S02 T04 D80*	
7	U007	100	K	S01 S02 T04 D80*	
8	U008	100	K	S01 S02 T04 D80*	
9	U009	100	K	S01 S02 T04 D80*	
10	U010	100	K	S01 S02 T04 D80*	
11	U011	100	K	S01 S02 T04 D80*	
12	U012	100	K	S01 S02 T04 D80*	
13	U014	100	K	S01 S02 T04 D80*	
14	U015	100	K	S01 S02 T04 D80*	
15	U016	100	K	S01 S02 T04 D80*	
16	U017	100	K	S01 S02 T04 D80*	
17	U018	100	K	S01 S02 T04 D80*	
18	U020	100	K	S01 S02 T04 D80*	
19	U021	50	K	S01 S02 T04 D80*	
20	U024	100	K	S01 S02 T04 D80*	
21	U025	100	K	S01 S02 T04 D80*	
22	U037	100	K	S01 S02 T04 D80*	
23	U038	100	K	S01 S02 T04 D80*	
24	U029	100	K	S01 S02 T04 D80*	
25	U034	100	K	S01 S02 T04 D80*	
26	U049	100	K	S01 S02 T04 D80*	

EPA Form 3510-3 (6-80)

CONTINUE ON REVERSE

*Additionally, Process Codes of T01, T02, S04

EPA I.D. NUMBER (enter from page 1): WPRD091018622 2

FOR OFFICIAL USE ONLY: W DUP DUP

IV. 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	U030	100	K	S01 S02 T04 D80*	
2	U025	100	K	S01 S02 T04 D80*	
3	U028	100	K	S01 S02 T04 D80*	
4	U023	100	K	S01 S02 T04 D80*	
5	U022	100	K	S01 S02 T04 D80*	
6	U036	100	K	S01 S02 T04 D80*	
7	U026	100	K	S01 S02 T04 D80*	
8	U043	100	K	S01 S02 T04 D80*	
9	U042	100	K	S01 S02 T04 D80*	
10	U045	100	K	S01 S02 T04 D80*	
11	U046	100	K	S01 S02 T04 D80*	
12	U047	100	K	S01 S02 T04 D80*	
13	U044	3,000	K	S01 S02 T04 D80*	
14	U059	100	K	S01 S02 T04 D80*	
15	U055	100	K	S01 S02 T04 D80*	
16	U058	100	K	S01 S02 T04 D80*	
17	U056	100	K	S01 S02 T04 D80*	
18	U067	100	K	S01 S02 T04 D80*	
19	U068	100	K	S01 S02 T04 D80*	
20	U069	100	K	S01 S02 T04 D80*	
21	U063	100	K	S01 S02 T04 D80*	
22	U070	100	K	S01 S02 T04 D80*	
23	U071	100	K	S01 S02 T04 D80*	
24	U072	100	K	S01 S02 T04 D80*	
25	U073	100	K	S01 S02 T04 D80*	
26	U074	100	K	S01 S02 T04 D80*	

EPA Form 3510-3 (6-80)

CONTINUE ON REVERSE

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

*Additionally, Process Codes (... of T01, T02, S04

Form Approved OMB No. 158-030004

EPA I.D. NUMBER (enter from page 1): W P R D 0 9 1 0 1 8 6 2 2 | T/A | C 1

FOR OFFICIAL USE ONLY: W | DUP | T/A | C 2 | DUP

IV. 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	U075	100	K	S01 S02 T04 D80*	
2	U076	100	K	S01 S02 T04 D80*	
3	U078	100	K	S01 S02 T04 D80*	
4	U079	100	K	S01 S02 T04 D80*	
5	U077	100	K	S01 S02 T04 D80*	
6	U082	100	K	S01 S02 T04 D80*	
7	U089	100	K	S01 S02 T04 D80*	
8	U080	4,000	K	S01 S02 T04 D80*	
9	U083	100	K	S01 S02 T04 D80*	
10	U088	100	K	S01 S02 T04 D80*	
11	U084	100	K	S01 S02 T04 D80*	
12	U086	100	K	S01 S02 T04 D80*	
13	U085	100	K	S01 S02 T04 D80*	
14	U081	100	K	S01 S02 T04 D80*	
15	U087	100	K	S01 S02 T04 D80*	
16	U064	100	K	S01 S02 T04 D80*	
17	U066	100	K	S01 S02 T04 D80*	
18	U062	100	K	S01 S02 T04 D80*	
19	U060	100	K	S01 S02 T04 D80*	
20	U061	100	K	S01 S02 T04 D80*	
21	U093	100	K	S01 S02 T04 D80*	
22	U090	100	K	S01 S02 T04 D80*	
23	U098	100	K	S01 S02 T04 D80*	
24	U099	100	K	S01 S02 T04 D80*	
25	U092	100	K	S01 S02 T04 D80*	
26	U094	100	K	S01 S02 T04 D80*	

EPA Form 3510-3 (6-80)

CONTINUE ON REVERSE

* Additionally, Process Codes of To1, TO2, S04

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

NOTE: Unless you have more than 26 wastes to list, do not use this page. If you have more than 26 wastes to list, use additional pages.

Form Approved OMB No. 158-R0004

EPA I.D. NUMBER (enter from page 1)	FOR OFFICIAL USE ONLY
WPRD091018622 T/A C 1	W DUP T/A C 2 DUP

IV. 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	U091	100	K	S01 S02 T04 D80*	
2	U106	100	K	S01 S02 T04 D80*	
3	U105	100	K	S01 S02 T04 D80*	
4	U102	100	K	S01 S02 T04 D80*	
5	U107	100	K	S01 S02 T04 D80*	
6	U109	100	K	S01 S02 T04 D80*	
7	U111	100	K	S01 S02 T04 D80*	
8	U112	5,000	K	S01 S02 T04 D80*	
9	U113	100	K	S01 S02 T04 D80*	
10	U114	100	K	S01 S02 T04 D80*	
11	U115	100	K	S01 S02 T04 D80*	
12	U116	100	K	S01 S02 T04 D80*	
13	U117	100	K	S01 S02 T04 D80*	
14	U118	100	K	S01 S02 T04 D80*	
15	U119	100	K	S01 S02 T04 D80*	
16	U120	100	K	S01 S02 T04 D80*	
17	U121	100	K	S01 S02 T04 D80*	
18	U122	1,500	K	S01 S02 T04 D80*	
19	U123	100	K	S01 S02 T04 D80*	
20	U124	100	K	S01 S02 T04 D80*	
21	U108	100	K	S01 S02 T04 D80*	
22	U101	100	K	S01 S02 T04 D80*	
23	U103	100	K	S01 S02 T04 D80*	
24	U108	100	K	S01 S02 T04 D80*	
25	U110	100	K	S01 S02 T04 D80*	
26		100	K	S01 S02 T04 D80*	

EPA Form 3510-3 (6-80)

CONTINUE ON REVERSE

* Additionally, Process Codes (enter "A", "B", "C", etc. behind the "J" to identify photocopied pages) of T01, T02, S04

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

Form Approved OMB No. 158-080004

EPA I.D. NUMBER (enter from page 1)	FOR OFFICIAL USE ONLY
W P R D 0 9 1 0 1 8 6 2 2 — T/A C: 1	W DUP — T/A C: 2 DUP

IV. 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	U125	100	K	S01 S02 T04 D80*	
2	U126	100	K	S01 S02 T04 D80*	
3	U127	100	K	S01 S02 T04 D80*	
4	U128	100	K	S01 S02 T04 D80*	
5	U129	100	K	S01 S02 T04 D80*	
6	U130	100	K	S01 S02 T04 D80*	
7	U131	100	K	S01 S02 T04 D80*	
8	U132	100	K	S01 S02 T04 D80*	
9	U134	100	K	S01 S02 T04 D80*	
10	U138	500	K	S01 S02 T04 D80*	
11	U139	100	K	S01 S02 T04 D80*	
12	U140	100	K	S01 S02 T04 D80*	
13	U141	100	K	S01 S02 T04 D80*	
14	U142	100	K	S01 S02 T04 D80*	
15	U143	100	K	S01 S02 T04 D80*	
16	U144	50	K	S01 S02 T04 D80*	
17	U145	100	K	S01 S02 T04 D80*	
18	U146	100	K	S01 S02 T04 D80*	
19	U147	100	K	S01 S02 T04 D80*	
20	U148	100	K	S01 S02 T04 D80*	
21	U149	100	K	S01 S02 T04 D80*	
22	U150	100	K	S01 S02 T04 D80*	
23	U151	10,000	K	S01 S02 T04 D80*	
24	U152	100	K	S01 S02 T04 D80*	
25	U153	100	K	S01 S02 T04 D80*	
26	U154	10,000	K	S01 S02 T04 D80*	

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*Additionally, Process Codes of T01, T02, S04

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

CONTINUE ON REVERSE

EPA I.D. NUMBER (enter from page 1)	FOR OFFICIAL USE ONLY
WPRD091018622 T/A C 1	W DUP T/A C 2 DUP

IV. 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	U155	100	K	S01 S02 T04 D80*	
2	U156	100	K	S01 S02 T04 D80*	
3	U157	100	K	S01 S02 T04 D80*	
4	U158	100	K	S01 S02 T04 D80*	
5	U159	100	K	S01 S02 T04 D80*	
6	U161	100	K	S01 S02 T04 D80*	
7	U162	100	K	S01 S02 T04 D80*	
8	U163	100	K	S01 S02 T04 D80*	
9	U164	100	K	S01 S02 T04 D80*	
10	U165	100	K	S01 S02 T04 D80*	
11	U166	100	K	S01 S02 T04 D80*	
12	U167	100	K	S01 S02 T04 D80*	
13	U168	100	K	S01 S02 T04 D80*	
14	U169	100	K	S01 S02 T04 D80*	
15	U170	100	K	S01 S02 T04 D80*	
16	U171	100	K	S01 S02 T04 D80*	
17	U172	100	K	S01 S02 T04 D80*	
18	U173	100	K	S01 S02 T04 D80*	
19	U174	100	K	S01 S02 T04 D80*	
20	U175	100	K	S01 S02 T04 D80*	
21	U177	100	K	S01 S02 T04 D80*	
22	U178	100	K	S01 S02 T04 D80*	
23	U179	100	K	S01 S02 T04 D80*	
24	U180	100	K	S01 S02 T04 D80*	
25	U181	100	K	S01 S02 T04 D80*	
26	U182	100	K	S01 S02 T04 D80	

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*Additionally, Process Codes of T01, T02, S04

CONTINUE ON REVERSE

EPA I.D. NUMBER (enter from page 1): WPRD091018622 2

FOR OFFICIAL USE ONLY: W DUP 2 DUP

IV. 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	U183	100	K	S01 S02 T04 D80*	
2	U184	100	K	S01 S02 T04 D80*	
3	U185	100	K	S01 S02 T04 D80*	
4	U187	100	K	S01 S02 T04 D80*	
5	U188	5,000	K	S01 S02 T04 D80*	
6	U186	100	K	S01 S02 T04 D80*	
7	U190	100	K	S01 S02 T04 D80*	
8	U197	100	K	S01 S02 T04 D80*	
9	U193	100	K	S01 S02 T04 D80*	
10	U192	100	K	S01 S02 T04 D80*	
11	U200	100	K	S01 S02 T04 D80*	
12	U201	50	K	S01 S02 T04 D80*	
13	U202	100	K	S01 S02 T04 D80*	
14	U206	100	K	S01 S02 T04 D80*	
15	U207	100	K	S01 S02 T04 D80*	
16	U208	100	K	S01 S02 T04 D80*	
17	U203	100	K	S01 S02 T04 D80*	
18	U209	100	K	S01 S02 T04 D80*	
19	U210	1,500	K	S01 S02 T04 D80*	
20	U218	100	K	S01 S02 T04 D80*	
21	U213	100	K	S01 S02 T04 D80*	
22	U211	100	K	S01 S02 T04 D80*	
23	U214	100	K	S01 S02 T04 D80*	
24	U220	5,000	K	S01 S02 T04 D80*	
25	U225	100	K	S01 S02 T04 D80*	
26	U226	12,000	K	S01 S02 T04 D80*	

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CONTINUE ON REVERSE

*Additionally, Process Codes of T01, T02, S04

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

EPA I.D. NUMBER (enter from page 1)	FOR OFFICIAL USE ONLY
WPRD091018622 T/A C 1	W DUP T/A C 2 DUP

IV. 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	U223	100	K	S01 S02 T04 D80*	
2	U227	100	K	S01 S02 T04 D80*	
3	U222	100	K	S01 S02 T04 D80*	
4	U235	100	K	S01 S02 T04 D80*	
5	U234	100	K	S01 S02 T04 D80*	
6	U236	100	K	S01 S02 T04 D80*	
7	U238	100	K	S01 S02 T04 D80*	
8	U239	2,000	K	S01 S02 T04 D80*	
9	U237	100	K	S01 S02 T04 D80*	
10	U243	100	K	S01 S02 T04 D80*	
11	U247	100	K	S01 S02 T04 D80*	
12	U248	100	K	S01 S02 T04 D80*	
13	U249	100	K	S01 S02 T04 D80*	
14	U213	100	K	S01 S02 T04 D80*	
15	U216	100	K	S01 S02 T04 D80*	
16	U217	100	K	S01 S02 T04 D80*	
17	U219	100	K	S01 S02 T04 D80*	
18	U218	100	K	S01 S02 T04 D80*	
19	U221	100	K	S01 S02 T04 D80*	
20	U228	5,000	K	S01 S02 T04 D80*	
21	U227	100	K	S01 S02 T04 D80*	
22	U231	100	K	S01 S02 T04 D80*	
23	U232	100	K	S01 S02 T04 D80*	
24	U230	100	K	S01 S02 T04 D80*	
25	U244	100	K	S01 S02 T04 D80*	
26	U240	100	K	S01 S02 T04 D80*	

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CONTINUE IN REVERSE

*Additionally, Process Codes of T01, T02, S04

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

Form Approved OMB

EPA I.D. NUMBER (enter from page 1)		FOR OFFICIAL USE ONLY		
W P R D 0 9 1 0 1 8 6 2 2	1	W	DUP	2 DUP

IV. 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	P023	100	K	S01 S02 T04 D80*	
2	P002	100	K	S01 S02 T04 D80*	
3	P057	100	K	S01 S02 T04 D80*	
4	P058	100	K	S01 S02 T04 D80*	
5	P066	100	K	S01 S02 T04 D80*	
6	P001	100	K	S01 S02 T04 D80*	
7	P002	100	K	S01 S02 T04 D80*	
8	P003	100	K	S01 S02 T04 D80*	
9	P070	100	K	S01 S02 T04 D80*	
10	P004	100	K	S01 S02 T04 D80*	
11	P005	100	K	S01 S02 T04 D80*	
12	P006	100	K	S01 S02 T04 D80*	
13	P007	100	K	S01 S02 T04 D80*	
14	P008	100	K	S01 S02 T04 D80*	
15	P119	100	K	S01 S02 T04 D80*	
16	P010	100	K	S01 S02 T04 D80*	
17	P012	100	K	S01 S02 T04 D80*	
18	P011	100	K	S01 S02 T04 D80*	
19	P012	100	K	S01 S02 T04 D80*	
20	P038	100	K	S01 S02 T04 D80*	
21	P024	100	K	S01 S02 T04 D80*	
22	P077	100	K	S01 S02 T04 D80*	
23	P028	100	K	S01 S02 T04 D80*	
24	P042	100	K	S01 S02 T04 D80*	
25	P014	100	K	S01 S02 T04 D80*	
26	P015	100	K	S01 S02 T04 D80*	

EPA Form 3510-3 (6-80)

CONTINUE ON REVERSE

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

Additionally, Process Codes of T01, T02, S04

EPA I.D. NUMBER (enter from page)	FOR OFFICIAL USE ONLY
W P R D 0 9 1 0 1 8 6 2 2 1	W DUP 2 DUP

IV. 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 D1)
1	P068	100	K	S01 S02 T04 D80*	
2	P069	100	K	S01 S02 T04 D80*	
3	P034	100	K	S01 S02 T04 D80*	
4	P072	100	K	S01 S02 T04 D80*	
5	P073	100	K	S01 S02 T04 D80*	
6	P075	100	K	S01 S02 T04 D80*	
7	P076	100	K	S01 S02 T04 D80*	
8	P077	100	K	S01 S02 T04 D80*	
9	P078	100	K	S01 S02 T04 D80*	
10	P048	100	K	S01 S02 T04 D80*	
11	P020	100	K	S01 S02 T04 D80*	
12	P049	100	K	S01 S02 T04 D80*	
13	P109	100	K	S01 S02 T04 D80*	
14	P087	100	K	S01 S02 T04 D80*	
15	P051	100	K	S01 S02 T04 D80*	
16	P089	100	K	S01 S02 T04 D80*	
17	P094	100	K	S01 S02 T04 D80*	
18	P020	100	K	S01 S02 T04 D80*	
19	P081	100	K	S01 S02 T04 D80*	
20	P101	100	K	S01 S02 T04 D80*	
21	P110	100	K	S01 S02 T04 D80*	
22	P054	100	K	S01 S02 T04 D80*	
23	P102	100	K	S01 S02 T04 D80*	
24	P111	100	K	S01 S02 T04 D80*	
25	P107	100	K	S01 S02 T04 D80*	
26	P108	100	K	S01 S02 T04 D80*	

EPA Form 3510-3 (6-80)

CONTINUE IN REVERSE

*Additionally, Process Codes of T01, T02, S04

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

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

Form Approved OMB No. 158-080004

EPA I.D. NUMBER (enter from page 1)		FOR OFFICIAL USE ONLY	
W P R D 0 9 1 0 1 8 6 2 2	T/A C 1	W DUP	T/A C 2 DUP

IV. 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	P016	100	K	S01 S02 T04 D80*	
2	P017	100	K	S01 S02 T04 D80*	
3	P123	100	K	S01 S02 T04 D80*	
4	P103	100	K	S01 S02 T04 D80*	
5	P056	100	K	S01 S02 T04 D80*	
6	P026	100	K	S01 S02 T04 D80*	
7	P027	100	K	S01 S02 T04 D80*	
8	P036	100	K	S01 S02 T04 D80*	
9	P037	100	K	S01 S02 T04 D80*	
10	P057	100	K	S01 S02 T04 D80*	
11	P041	100	K	S01 S02 T04 D80*	
12	P040	100	K	S01 S02 T04 D80*	
13	P043	100	K	S01 S02 T04 D80*	
14	P044	100	K	S01 S02 T04 D80*	
15	P045	100	K	S01 S02 T04 D80*	
16	U019	100	K	S01 S02 T04 D80*	
17	U095	100	K	S01 S02 T04 D80*	
18	U097	100	K	S01 S02 T04 D80*	
19	U212	100	K	S01 S02 T04 D80*	
20	P118	100	K	S01 S02 T04 D80*	
21	P067	100	K	S01 S02 T04 D80*	
22	P105	100	K	S01 T01 D80	
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)

*Additionally, Process Codes of T01,T02,S04

IV. DESCRIPTION OF HAZARDOUS WASTES (continued)

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

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

F P R D 0 9 1 0 1 8 6 2 2 6

V. FACILITY DRAWING

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

VI. PHOTOGRAPHS

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

VII. FACILITY GEOGRAPHIC LOCATION

LATITUDE (degrees, minutes, & seconds)	LONGITUDE (degrees, minutes, & seconds)
18 01 05	66 41 03

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.
E Compania Ganadera del Sur, Inc.	801-836-1086

3. STREET OR P.O. BOX	4. CITY OR TOWN	5. ST.	6. ZIP CODE
F Firm Delivery	G Ponce	PR	00731

IX. OWNER CERTIFICATION

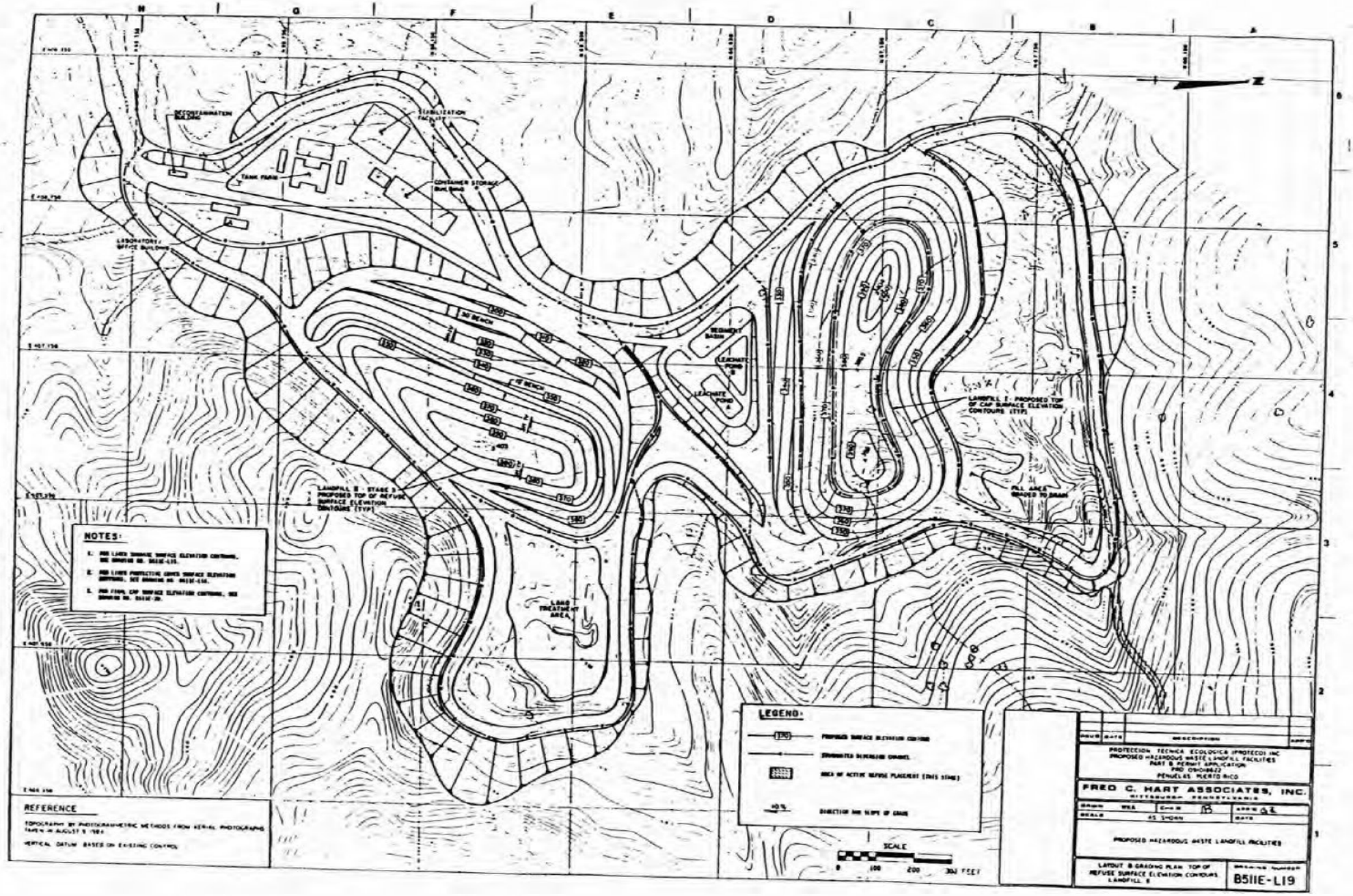
I certify under penalty of law that I have personally examined and am familiar with the information submitted in this and all attached documents, and that based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, 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
Lucas P. Valdivieso	Lucas P. Valdivieso	4-1-86

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
Jorge J. Fernandez	Jorge J. Fernández Porto	4-1-86



DECONTAMINATION BUILDING
STABILIZATION FACILITY
TANK FARM
CONTAINER STORAGE BUILDING
LABORATORY/ OFFICE BUILDING
30' BENCH
SEDIMENT BASIN
LEACHATE POND B
LEACHATE POND A
LANDFILL I - PROPOSED TOP OF CAP SURFACE ELEVATION CONTOURS (TYP)
FILL AREA GRADED TO DRAIN
LANDFILL II - STAGE 3 PROPOSED TOP OF REFUSE SURFACE ELEVATION CONTOURS (TYP)
LAND TREATMENT AREA
NOTES:
LEGEND:
PROPOSED SURFACE ELEVATION CONTOUR
STORMWATER DIVERSION CHANNEL
AREA OF ACTIVE REFUSE PLACEMENT (THIS STAGE)
DIRECTION AND SLOPE OF GRADE
SCALE
0 100 200 300 FEET
REFERENCE
TOPOGRAPHY BY PHOTOGRAMMETRIC METHODS FROM AERIAL PHOTOGRAPHS TAKEN IN AUGUST 5 1984
VERTICAL DATUM BASED ON EXISTING CONTROL
PROTECCION TECNICA ECOLOGICA (PROTECO) INC
PROPOSED HAZARDOUS WASTE LANDFILL FACILITIES
PART B PERMIT APPLICATION
PENUELAS, PUERTO RICO
FRED C. HART ASSOCIATES, INC.
PITTSBURGH PENNSYLVANIA
DRAWN WKA
SCALE AS SHOWN
PROPOSED HAZARDOUS WASTE LANDFILL FACILITIES
LAYOUT & GRADING PLAN TOP OF REFUSE SURFACE ELEVATION CONTOURS LANDFILL II
DRAWING NUMBER
B511E-L19

APPENDIX B.1

RIGHT OF WAY APPLICATION

SECTION B - FACILITY DESCRIPTION

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SECTION B

FACILITY DESCRIPTION

This section provides a general description of the hazardous waste management facility operated by Proteccion Tecnica Ecologica, Inc., PROTECO, a topographic map of the present facility and proposed extensions, and facility location and traffic pattern information. This description is intended to acquaint the permit application reviewer with an overview of the facility. More complete details can be found in other parts of this permit application. This information is submitted in accordance with 40 CFR 270.14(b)(1), (10), (11), (19) and the Puerto Rico Environmental Quality Board's Regulations for the Control of Hazardous and Non-Hazardous Solid Wastes, Part IX, Rule 902.

B-1 General Description [40 CFR 270.14(b)(1)]

The mailing address is:

Proteccion Tecnica Ecologica, Inc.
Firm Delivery
Ponce, Puerto Rico 00731

The activities conducted at the PROTECO hazardous waste management facility include the treatment, storage and disposal of hazardous waste and the treatment and disposal of non-hazardous waste. The hazardous wastes received and accepted at the facility are generated from many diverse manufacturing and government operations located in the Commonwealth of Puerto Rico. The hazardous waste is transported from these generators to the facility primarily by the PROTECO transportation staff. Table B-1 lists many of the types of industries served by PROTECO, the products they produce and the hazardous wastes generated from the manufacture of these products.

TABLE B-1

TYPE OF INDUSTRIES SERVICED

SIC Code	Typical Manufactured Products	Typical Wastes Generated
2834	Antibiotics, Pharmaceutical Products	F001
3679	Magnetic Recording Heads	F002
3471	Metal Finishing-Electroplating	D011
2834	Health Care Products-Thermometers	U151
2911	Petroleum Refining	D008
3679, 3573	Printed Circuits Manufacturing and Computer Products	F001, F006, D008, D002, D001
2653	Corrugated Boxes	D008
2834	Pharmaceutical Products, Laxat	)03)09,
2819	Diagnostic Chemical Reagents	
2067	Chewing Gum	
3843	Dental Floss	D001
3841	Membrane Filters & Associated Devices	D001
2911, 5172	Petroleum R	D008
3662, 3666	Communicati	D001, F001 F002
3662, 3651	Two-way Rad	D001, F001
3842	Disposable	D001, F002, F005
3811	High Pressure Chromotography Systems	D001
3622	Line Starters, Magnetic Contractor Relays	F002, D008

Give names of manufacturers (major clients) possible PRP's

What about the K, P and U wastes listed in the Part B?

(0012c)

Two methods are currently used to treat the hazardous wastes accepted for treatment, storage and disposal to render the wastes less hazardous, non-hazardous, stabilized and/or more amenable to disposal. The primary method used is stabilization/fixation. This treatment procedure renders a waste to a concrete-like solid material by reacting lime, fly ash, cement kiln dust, water and waste to form the solid. These treatment methods are discussed in more detail in the following sections. PROTECO also employs neutralization processes. Neutralization is a common treatment practice used to adjust the pH of a waste to an acceptable pH level (6.0 to 9.0) by combining acidic and alkaline materials.

Table B-2 summarizes the proposed treatment, storage and disposal practices at Proteco.

Figure B-1 locates these proposed treatment, storage and disposal sites at the PROTECO facility. The proposed tank farm consists of the following hazardous waste management units:

- Seven aboveground storage tanks, ID Nos. T1, T2, T4, T5, T6, T7 and T8,
- One aboveground treatment (neutralization) tank, ID. No. T3.

The facility accepts a wide range of hazardous wastes. PROTECO treats, stores and disposes of those wastes that are liquid, solid, sludges or slurries. Both characteristic and listed hazardous wastes as defined by 40 CFR 261 are accepted for treatment, storage and disposal. These include but are not limited to:

- Ignitable wastes
- Corrosive waste
- EP toxic wastes
- Halogenated solvents
- Pesticide waste
- Electroplating sludges

TABLE B-2

SUMMARY OF PROPOSED TREATMENT, STORAGE AND DISPOSAL PRACTICES AT PROTECO

STORAGE FACILITIES

Container Storage Facility

Surface Impoundments

Tanks

T-1 Alkali Tank
T-2 Acid Tank
T-4 Halogenated Solvents Tank
T-5 Non-Halogenated Solvents Tank
T-6 Oil Sludge Tank
T-7 Aqueous Tank
T-8 Oils Tank

TREATMENT FACILITIES

Tank

T-3 Neutralization Tank

Stabilization/Fixation Unit

DISPOSAL FACILITIES

Landfill I
Landfill II

FIGURE B-1

IDENTIFICATION OF FACILITIES AT PROTECO SITE

This topographic map can be found in a map pocket at the end of the PROTECO RCRA Part B Permit Application, Volume 1.

B-2 Topographic Map [40 CFR 270.14(b)(19)]

Figure B-1 is a topographic map of the hazardous waste facility. This map provides a layout of the facility with relief greater than 6.1 meters (20 feet), for which the regulation normally seeks a 1.5 meter (5 feet) contour interval. The regulation also suggests that the owner of facilities located in mountainous areas should use a larger contour interval. At this site the relief is 50 meters (164 feet) and the contour interval of two feet has been chosen to clearly show the pattern of surface water flow in the vicinity of and from each operational unit of the facility. Four foot contours are provided in steeper areas surrounding the waste management area due to sharply rising topographical relief.

B-2a Map Scale and Date

Figure B-1 identifies the location of proposed facilities at the PROTECO site. Figure B-1 is provided with a scale of 1 to 1,200 (one inch to 100 feet) from base topography developed in August 9, 1984. Figure B-1 also provides additional topographical information of the area 1,000 feet around the site. 1 2

B-2b 100-Year Floodplain Area

The Flood Insurance Rate Map for Puerto Rico, Community Panel Number 720000, Panel 0218B is shown as Figure B-2. This map demonstrates that the facility is not in a 100-year floodplain. 2

The Tallaboa River and its tributaries originate on the southern slope of the Central Cordillera and flow southward into the Tallaboa Bay. The Lower Tallaboa Valley, for approximately four miles, is entrenched and confined by rugged upland that borders both the east and west flanks of the valley. The steep bluff rises steeply over forty meters from the adjacent relatively flat river valley. Thus, flood waters, even the 100-year flood plain, remain confined or restricted to the entrenched

FIGURE B-2

FLOOD INSURANCE RATE MAP

This map can be found in a map pocket at the end of the PROTECO RCRA Part B Permit Application, Volume 1.

valley proper and some of the indented ravines along the cliff's flanks. PROTECO's facilities are located 70 meters above the maximum water level a 100-year flood could generate (Figure B-3).

B-2c Surface Water

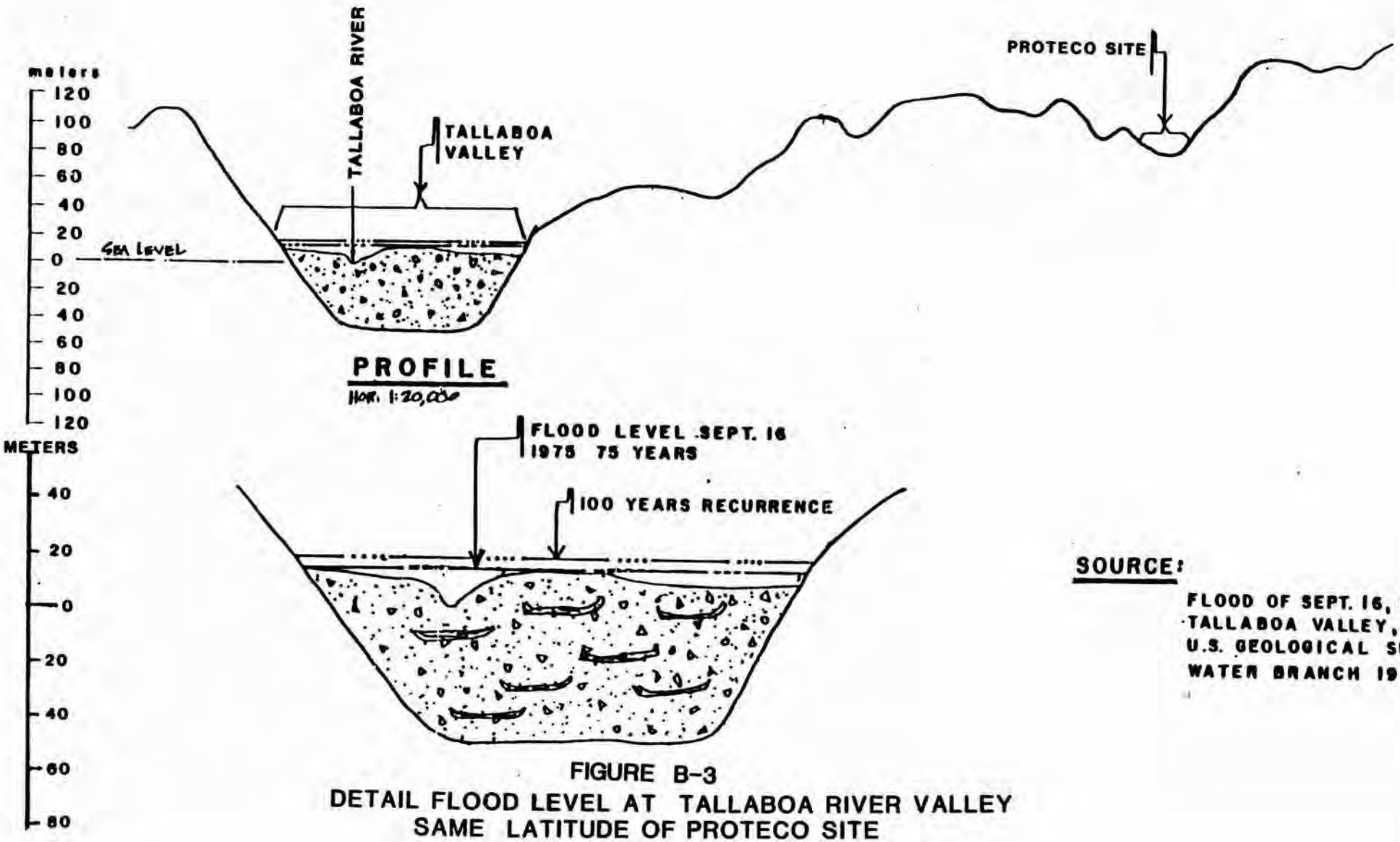
Presently a small natural channel carved by an intermittent brook drains the area only during extraordinary rainfall. No perennial flow exists, otherwise, along this channel which lies on a topographical lower level from the main facilities. The main drainage channel is 3 feet wide and 2 feet deep at its maximum and it is entrenched in case hardened surface of calcareous material located outside of PROTECO's property. The channel runs from northeast to southwest along near the site and ultimately drains into the Rio Tallaboa.

A surface water management project has been designed to control any surface flows over and around the site. Calculations used in developing this system are given in Appendix D-6.5.

B-2d Land Use

The land surrounding the site is primarily unused due to the remote location and steep terrain. Grazing of a "cebu" breed cattle takes place on a limited scale in areas located 2,000 - 3,000 meters southeast of the site. The scant rainfall and rugged calcareous geological setting has precluded agricultural activity in the region. There are no man made structures for over 1000 meters in any direction except for electric power transmission lines which cross the north end of the site. PROTECO has applied to Puerto Rico Electrical Power Authority (PREPA) for right of way along these power lines. The drawings and accompanying memo for this application are in Appendix B.1.

There are also no permanent residences for over 2.3 kilometers in any direction. Because of the rugged and inhospitable terrain there are no plans for land development in the vicinity of the site.



SOURCE:

FLOOD OF SEPT. 16, 1975
TALLABOA VALLEY, P.R.
U.S. GEOLOGICAL SURVEY
WATER BRANCH 19

FIGURE B-3
DETAIL FLOOD LEVEL AT TALLABOA RIVER VALLEY
SAME LATITUDE OF PROTECO SITE

On the other hand, the favorable coastal geographical and topographical location along the lower reaches of the Tallaboa Valley, together with the natural harbor existing adjacent to these coastal reaches, have made the area desirable for petrochemical complexes, power generating plant and allied industries. PROTECO's waste-management site is located 3.5 kilometers north of the Caribbean seashore and 2.5 kilometers immediately east of the Tallaboa River Valley (as noted in Figure B-4).

B-2e Wind Rose

Figure B-5 shows the annual average prevailing windspeed and direction. This figure indicates that approximately 70 percent of the annual wind is in an easterly direction, away from the closest communities (Seboro and Tallaboa).

B-2f Orientation

The north arrow is shown on the map, Figure B-1.

B-2g Legal Boundaries

The PROTECO facility is presently being surveyed to establish the legal boundaries of all hazardous waste units and operations. The results of this survey will be submitted to the USEPA and the EQB as an Addendum to this Part B upon finalization.

B-2h Access Control

The facility is surrounded by fences and/or natural barriers. Access control is discussed in further detail in Section F-1. Only one road, whose access is controlled, connects the facilities with major highways. The confinement and isolation of the site is more accentuated by the fact that the inner valley is surrounded by high-relief steep-sloping hills which are fully covered with xerophilous vegetation all year round. This inhospitable topographical setting and vegetative growth acts also as a natural barrier.

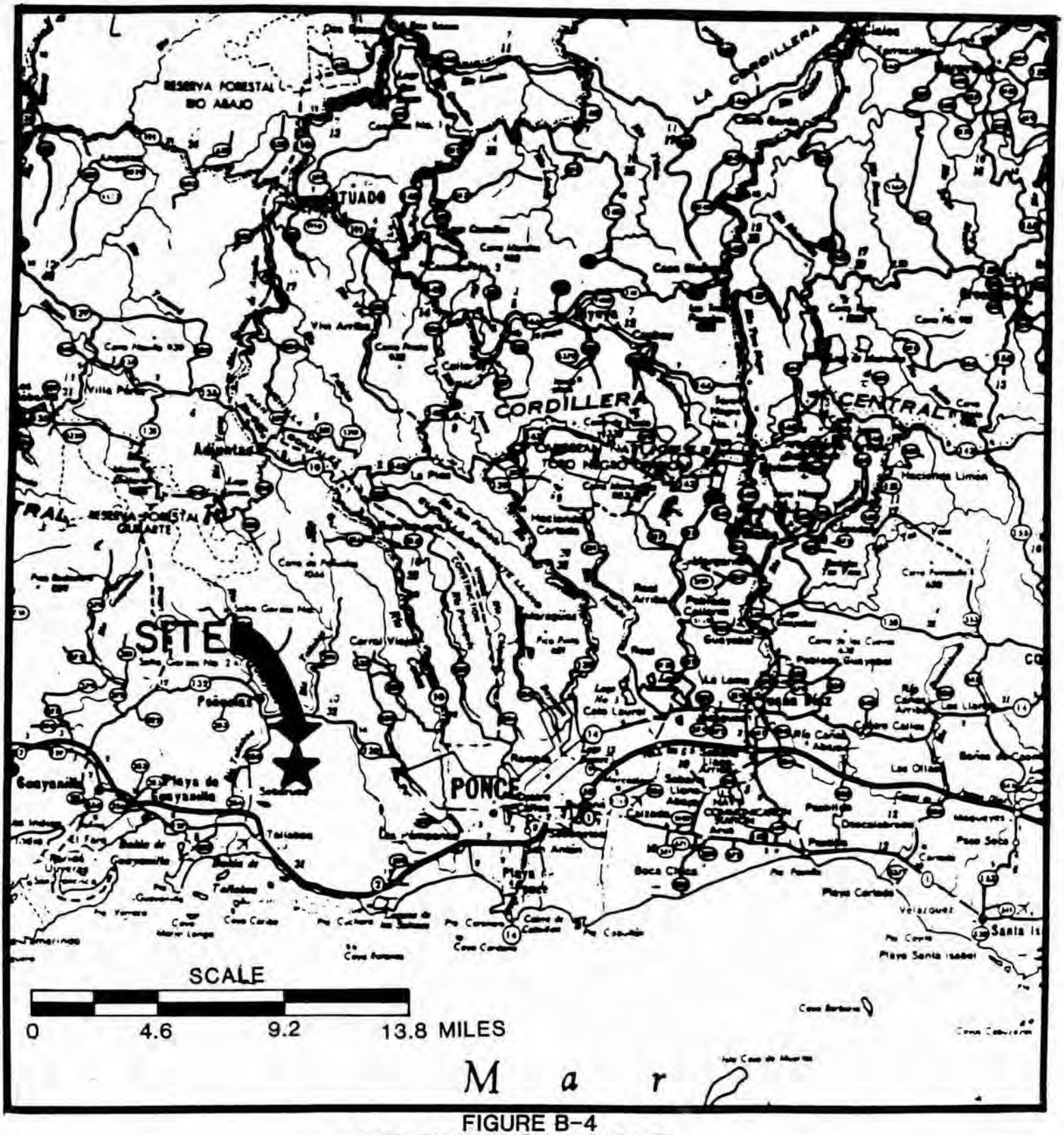


FIGURE B-4

LOCATION MAP

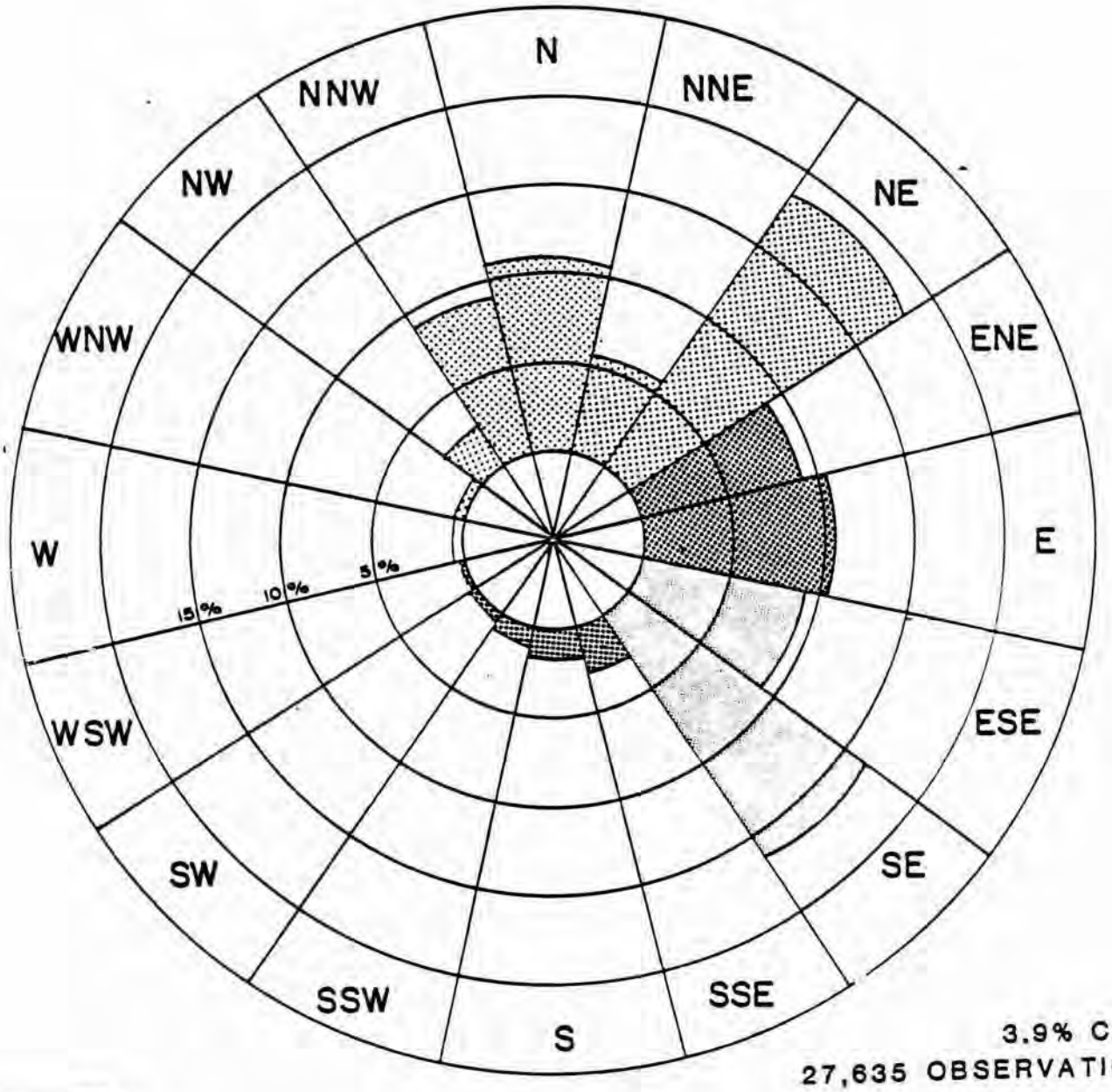
REFERENCE:

PUERTO RICO ROAD MAP,
DATED 1979, SCALE: 1" = 4.6 MILES

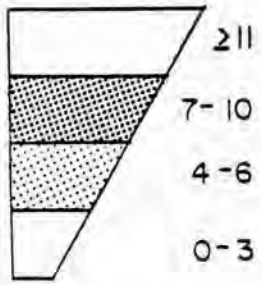
FIGURE B-5

WIND ROSE

SHOWING AVERAGE ANNUAL WINDSPEED AND DIRECTION



3.9% CALM
27,635 OBSERVATIONS



AVG. WIND SPEED, mph

PONCE, PUERTO RICO
LOSEY ARMY AIR FIELD
1941-1944

A new chain-link fence with a electric gate at the only entrance to the facility is shown in Figure B-1. Two locked evacuation gates are located along the west side of the facility. In addition, the stabilization/fixation unit, the container storage facility and the tank storage/treatment unit are surrounded by a six-foot, chain link fence. A five-foot, four strand barbed wire fence surrounds the entire facility. 1

B-2i Injection and Withdrawal Wells

The site has no injection wells. There are monitoring wells on the site which are not used for the withdrawal of water. There are no injection wells and there are no withdrawal wells off-site within 305 m (1,000 feet) of the facility.

B-2j Proposed Buildings, Treatment, Storage or Disposal Operations

Planned structures are shown on Figure B-1.

Treatment, Storage and Disposal Operations. The proposed tank storage area is shown on Figure B-1. Locations of the proposed landfills are also shown on Figure B-1. Engineering drawings for the Container Storage Facility, Tank Storage/Treatment Area, and Landfills I and II have been submitted to the EPA.

Run-off Control Systems. The design of the Run-off control systems is provided in Appendix D-6.4. 1

Internal Roads. These roads are shown in Figure B-1.

Sewers. None are proposed.

Loading and Unloading Areas. A concrete loading/unloading hardstand is provided at the Container Storage Facility. Other loading and unloading areas are located on Figure B-1.

Fire Control Facilities. PROTECO will upgrade present fire control programs as described in Section F.

B-2k Flood Control/Drainage Barrier

Due to the sites height above the flood plain, there is no need for a flood control barrier for off-site flooding conditions. As part of this Application, a comprehensive stormwater run-on control system has been designed to safely carry stormwater away from all regulated units. This system consists of thousands of feet of riprap-lined drainage-ways, piping and roadway crossings to carry the required design storm run-off around the units to a point downgradient of the unit at the lowest surface perimeter elevation, in this case, the tank farm. From this point, run-off is conveyed by naturally topography down the access road to the facility, as shown on Figure B-6. 2 1

B-3 Location Information [40 CFR 270.14(b)(11)]

B-3a Seismic Standard [40 CFR 270.14(b)(11)(i) and (ii)]

Because this facility is not listed in Appendix VI of Part 264, no further information is required to demonstrate compliance with §264.18(a). Seismic activity in this area is not significant for the purposes of this permit.

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

This facility is not located within a 100-year floodplain. The facility is in Zone 'c', Not Floodable. The source of information is the Flood Insurance Rate Map for Puerto Rico, Community Panel Number 720000, Panel 0218B effective July 2, 1981. This map, Figure B-2, can be found in a map pocket at the end of Volume 1 of this application. 2

B-4 Traffic Patterns [40 CFR 270.14(b)(10)]

Access to PROTECO is from P.R. 2 to P.R. 385 to the private access road leading directly to the site. P.R. 2 is a four-lane, all weather, hard surface, primary highway. The P.R. 2/P.R. 385 intersection is a modified cloverleaf, from which there is immediate (less than 0.8 Km or 1

VICINITY MAP

FIGURE B-6

REFERENCE:

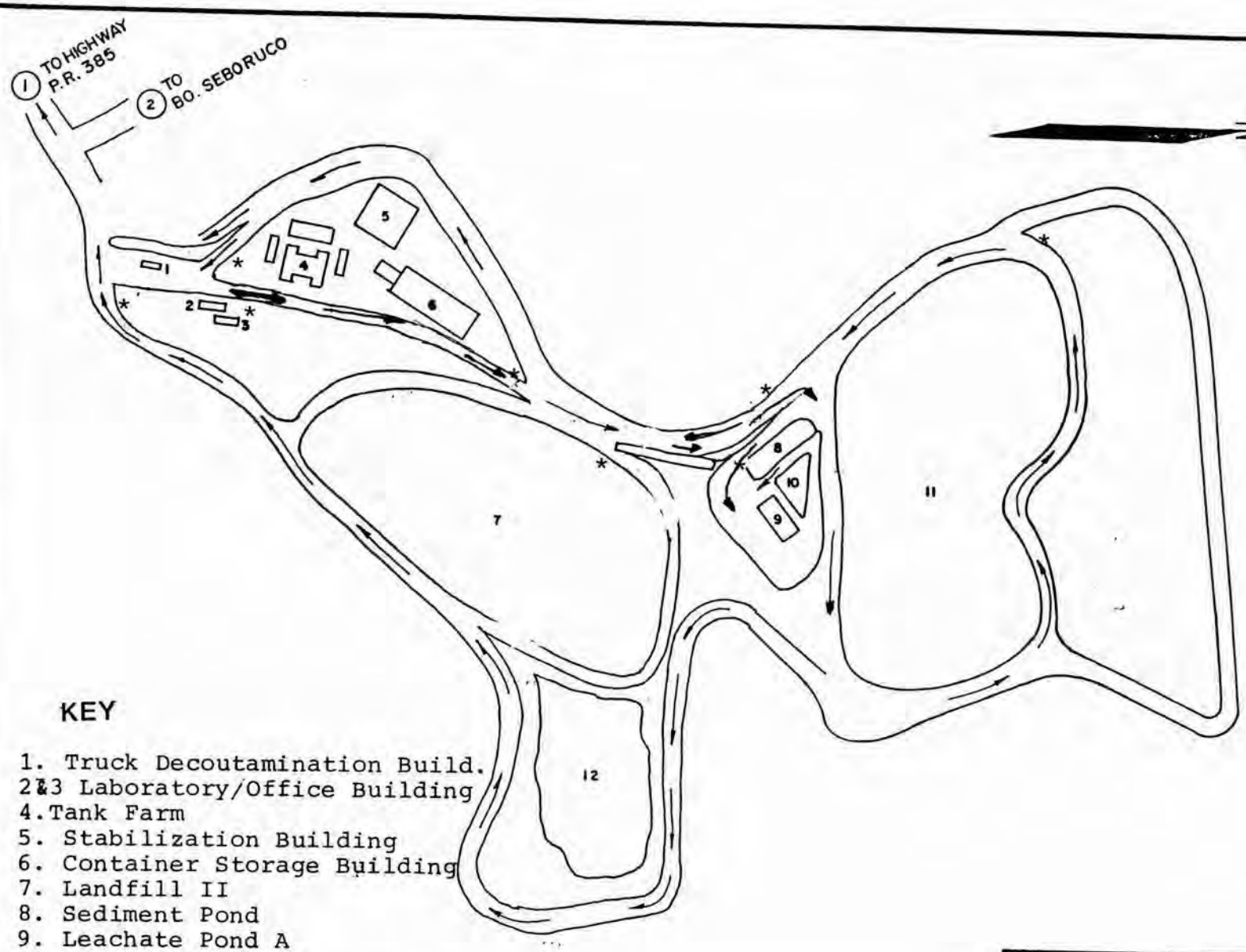
PENUELAS, P.R.
N1800-W6637.5/7.5
1972
PHOTOREVISED 1982
DMA 122211 NW-SERIES E835

SCALE

0 2000 4000 6000 FEET

Traffic Control. Stop signs will be used to control all return traffic flow from dead end roads. Stop signs will also control all traffic flow entering and leaving access/exit gates. In addition yellow painted cement highway markers are installed on roadways throughout the site. Figure B-7 shows the routes of hazardous waste vehicles on-site, along with all traffic control signs at the facility.

The basic traffic pattern on-site will initiate with a stop at the entrance gate, where the guard will direct the driver to the wasteload acceptance area, directly next to the laboratory facility. After the waste load is accepted at the facility, the truck is directed to proceed down the main road to the stabilization/fixation facility, tank farm, container storage facility or landfill. The truck returns to the gate along the western road, though the truck decontamination pad prior to exiting.



KEY

1. Truck Decoutamination Build.
2&3 Laboratory/Office Building
4. Tank Farm
5. Stabilization Building
6. Container Storage Building
7. Landfill II
8. Sediment Pond
9. Leachate Pond A
10. Leachate Pond B
11. Landfill I
12. Existing Land Treatment Area

* Stop Sign

FIGURE B-7
PROPOSED FACILITIES
TRAFFIC ROUTES

ENGINEERING, ARCHITECTURAL AND PLANNING CONSULTANTS

LEBRON ASSOCIATES

Date June 20-1986

Ref Protoco Bldgs. & Site

TO: PREPA

ATTN: Eng. Máximo Báez

WE ARE TRANSMITTING:

- [x] herewith
- [ ] under separate cover
- [x] by messenger
- [ ] air mail
- [ ] regular mail
- [ ] other

- [ ] REPORT
- [ ] DETAILS
- [ ] SPECIFICATIONS
- [ ] COST ESTIMATE
- [ ] REPRODUCIBLES
- [ ] INVOICE
- [x] DRAWINGS/BLUEPRINTS
- [ ] COPY OF
- [ ] SETS OF PLANS
- [ ] APPROVED
- [ ] DISAPPROVED
- [ ] AS REQUESTED
- [ ] YOUR INFORMATION
- [x] YOUR COMMENTS
- [ ] YOUR APPROVAL
- [ ] RETURN TO SENDER
- [ ] DISTRIBUTION
- [ ] YOUR FILE

REMARKS:

A copy of electric line profile & electric site plans for approval of landuse underneath the line.

Please Sign and Return Promptly Enclosed Copy.

LEBRON ASSOCIATES

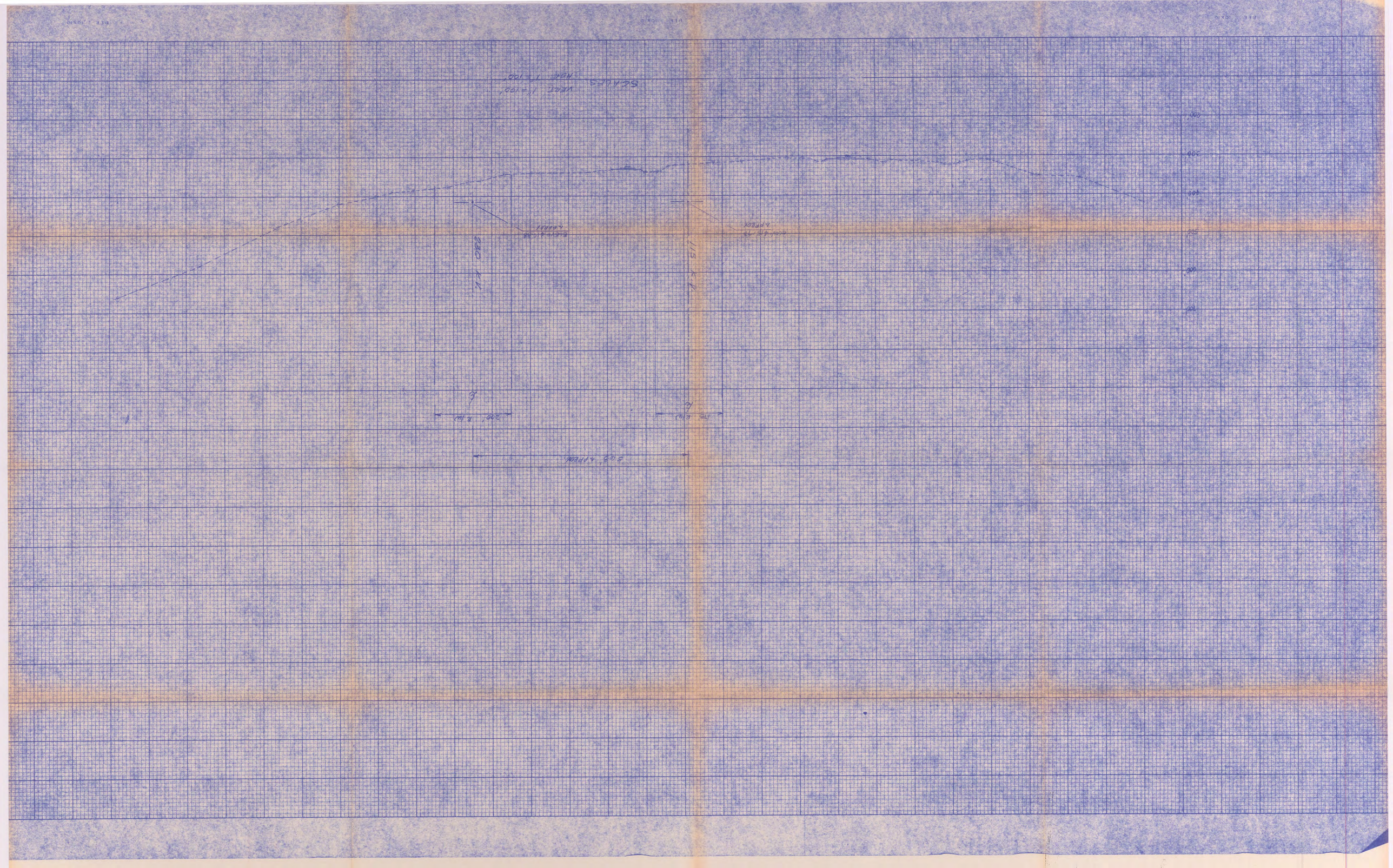
By: _____

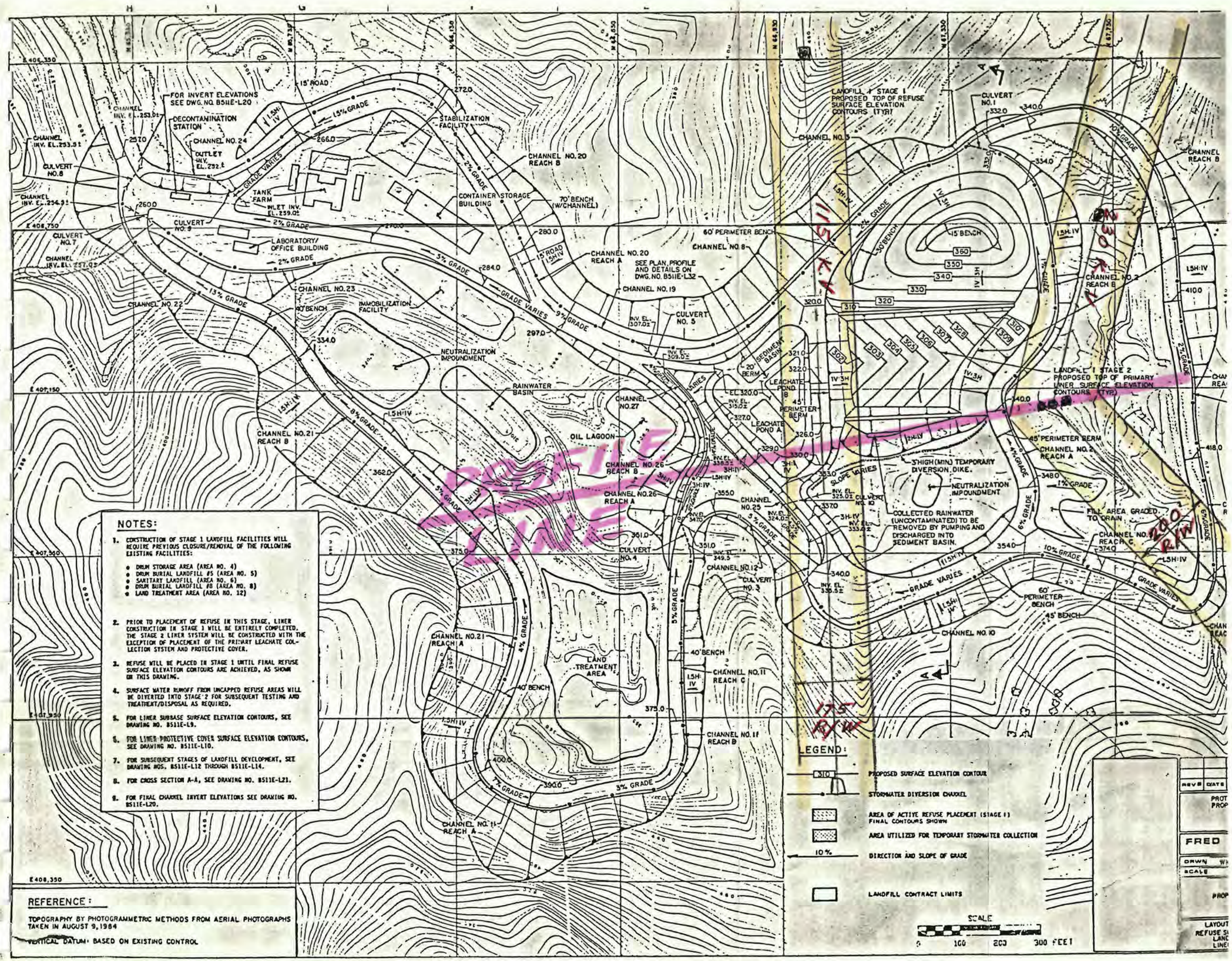
Received _____

By _____

Date _____

P.O. BOX H.J., CAPARRA HEIGHTS STATION, SAN JUAN, PUERTO RICO 00922 / TEL.: (809) 723-3332 / CABLE: LEBRASS
1 MIRAFLORES STREET, MIRAMAR, PUERTO RICO


SCALES
VERT 1"=100'
HOR 1"=100'
230 KV
115 KV
200' R/W
175' R/W
5 to 8' APPROX.


FOR INVERT ELEVATIONS SEE DWG. NO. B511E-L20
DECONTAMINATION STATION
CHANNEL NO. 24
OUTLET INV. EL. 232.±
CHANNEL INV. EL. 253.0±
CHANNEL INV. EL. 253.5±
CULVERT NO. 8
CHANNEL INV. EL. 256.5±
CULVERT NO. 7
CHANNEL INV. EL. 257.0±
15' ROAD
15% GRADE
GRADE VARIES
TANK FARM
INLET INV. EL. 259.0±
2% GRADE
CULVERT NO. 9
LABORATORY/ OFFICE BUILDING
13% GRADE
CHANNEL NO. 22
CHANNEL NO. 23
60' BENCH
IMMOBILIZATION FACILITY
NEUTRALIZATION IMPOUNDMENT
RAINWATER BASIN
CHANNEL NO. 21 REACH B
8% GRADE
STABILIZATION FACILITY
CHANNEL NO. 20 REACH B
CONTAINER STORAGE BUILDING
70' BENCH (W/CHANNEL)
CHANNEL NO. 20 REACH A
SEE PLAN, PROFILE AND DETAILS ON DWG. NO. B511E-L32
CHANNEL NO. 19
CULVERT NO. 5
9% GRADE
CHANNEL NO. 27
OIL LAGOON
CHANNEL NO. 26 REACH B
CHANNEL NO. 26 REACH A
CHANNEL NO. 25
CULVERT NO. 4
CHANNEL NO. 12
CULVERT NO. 3
CHANNEL NO. 21 REACH A
LAND TREATMENT AREA
40' BENCH
CHANNEL NO. 11 REACH C
CHANNEL NO. 11 REACH B
CHANNEL NO. 11 REACH A
3% GRADE
60' PERIMETER BENCH
CHANNEL NO. 8
LANDFILL 1 STAGE 1 PROPOSED TOP OF REFUSE SURFACE ELEVATION CONTOURS (TYP.)
CULVERT NO. 1
CHANNEL NO. 5
15' BENCH
10' BENCH
CHANNEL NO. 2 REACH B
SEDIMENT BASIN
20' BERM
LEACHATE POND B
45' PERIMETER BERM
LEACHATE POND A
LANDFILL 1 STAGE 2 PROPOSED TOP OF PRIMARY LINER SURFACE ELEVATION CONTOURS (TYP.)
65' PERIMETER BERM
CHANNEL NO. 2 REACH A
3' HIGH (MIN.) TEMPORARY DIVERSION DIKE.
NEUTRALIZATION IMPOUNDMENT
COLLECTED RAINWATER (UNCONTAMINATED) TO BE REMOVED BY PUMPING AND DISCHARGED INTO SEDIMENT BASIN.
FILL AREA GRADED TO DRAIN
CHANNEL NO. 1 REACH C
10% GRADE
CHANNEL NO. 10
60' PERIMETER BENCH
45' BENCH
SLOPE VARIES
4% GRADE
6% GRADE
1% GRADE
CHANNEL NO. 2 REACH B
CHANNEL NO. 2 REACH A
E 406,350
E 406,750
E 407,150
E 407,550
E 407,950
E 408,350
NOTES:
1. CONSTRUCTION OF STAGE 1 LANDFILL FACILITIES WILL REQUIRE PREVIOUS CLOSURE/REMOVAL OF THE FOLLOWING EXISTING FACILITIES:
• DRUM STORAGE AREA (AREA NO. 4)
• DRUM BURIAL LANDFILL #5 (AREA NO. 5)
• SANITARY LANDFILL (AREA NO. 6)
• DRUM BURIAL LANDFILL #8 (AREA NO. 8)
• LAND TREATMENT AREA (AREA NO. 12)
2. PRIOR TO PLACEMENT OF REFUSE IN THIS STAGE, LINER CONSTRUCTION IN STAGE 1 WILL BE ENTIRELY COMPLETED. THE STAGE 2 LINER SYSTEM WILL BE CONSTRUCTED WITH THE EXCEPTION OF PLACEMENT OF THE PRIMARY LEACHATE COLLECTION SYSTEM AND PROTECTIVE COVER.
3. REFUSE WILL BE PLACED IN STAGE 1 UNTIL FINAL REFUSE SURFACE ELEVATION CONTOURS ARE ACHIEVED, AS SHOWN ON THIS DRAWING.
4. SURFACE WATER RUNOFF FROM UNCAPPED REFUSE AREAS WILL BE DIVERTED INTO STAGE 2 FOR SUBSEQUENT TESTING AND TREATMENT/DISPOSAL AS REQUIRED.
5. FOR LINER SUBBASE SURFACE ELEVATION CONTOURS, SEE DRAWING NO. B511E-L9.
6. FOR LINER PROTECTIVE COVER SURFACE ELEVATION CONTOURS, SEE DRAWING NO. B511E-L10.
7. FOR SUBSEQUENT STAGES OF LANDFILL DEVELOPMENT, SEE DRAWING NOS. B511E-L12 THROUGH B511E-L14.
8. FOR CROSS SECTION A-A, SEE DRAWING NO. B511E-L21.
9. FOR FINAL CHANNEL INVERT ELEVATIONS SEE DRAWING NO. B511E-L20.
REFERENCE:
TOPOGRAPHY BY PHOTOGRAMMETRIC METHODS FROM AERIAL PHOTOGRAPHS TAKEN IN AUGUST 9, 1984
VERTICAL DATUM: BASED ON EXISTING CONTROL
LEGEND:
PROPOSED SURFACE ELEVATION CONTOUR
STORMWATER DIVERSION CHANNEL
AREA OF ACTIVE REFUSE PLACEMENT (STAGE 1) FINAL CONTOURS SHOWN
AREA UTILIZED FOR TEMPORARY STORMWATER COLLECTION
DIRECTION AND SLOPE OF GRADE
LANDFILL CONTRACT LIMITS
SCALE
0 100 200 300 FEET
REVS DATE
FRED
DRWN
SCALE
LAYOUT
REFUSE SI
LANC
LINE
175' R/W
100' R/W

SECTION C - WASTE CHARACTERISTICS AND ANALYSIS PLAN

SECTION C

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SECTION C

WASTE CHARACTERISTICS

This section describes the chemical and physical nature of the hazardous wastes treated, stored or disposed of at Proteccion Tecnica Ecologica, Inc. (PROTECO); the waste analysis plan for sampling, testing, and evaluating wastes to assure that sufficient information is available to treat, store and dispose of the waste; the screening and evaluation of each waste shipment before acceptance at the facility and the trial testing program for treatment of wastes by stabilization/fixation.

This section describes the proposed waste analysis program that is to be implemented by PROTECO. This information is submitted in accordance with the requirements of 40 CFR 270.14(b)(2) and (3) and the Puerto Rico Environmental Quality Board (EQB) Regulations, Part VIII - Rule 807I and Part IX - Rule 902D.

C-1 Chemical and Physical Analyses [40 CFR 270.14(b)(2)]

C-1a General Disposition of Hazardous Wastes Treated, Stored or Disposed of at the Facility

Proposed facilities at PROTECO include a container storage facility, a tank storage area with eight tanks providing a total of 160,000 gallons of storage and treatment, and two new landfill facilities with a combined design capacity of approximately 1,000,000 cubic yards. A list of the wastes reasonably anticipated to be treated, stored or disposed of at the PROTECO facility and the basis for the hazard designation is presented in Table C-1.

Hazardous wastes accepted at PROTECO will be handled in one of four general methods: storage in a tank or containerized waste area, neutralization, stabilization/fixation and/or secure landfilling. The primary method used to treat wastes is stabilization/fixation. Typically, up to one half of all the waste streams received by the facility will be treated by this method.

TABLE C-1

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (if available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
D001	Ignitable waste	Ignitable	Flash point
D002	Corrosive waste	Corrosive	pH
D004	Arsenic	EP Toxic	Arsenic
D005	Barium	EP Toxic	Barium
D006	Cadmium	EP Toxic	Cadmium
D007	Chromium	EP Toxic	Chromium
D008	Lead	EP Toxic	Lead
D009	Mercury	EP Toxic	Mercury
D010	Selenium	EP Toxic	Selenium
D011	Silver	EP Toxic	Silver
D012	Endrin	EP Toxic	1,2,3,4,10,10-hexachloro-1,7-epoxy=1,4,4a,5,6,7,8,8a-octahydro-1,4-endo, endo-5,8-dimethano napthalene
D013	Lindane	EP Toxic	1,2,3,4,5,6-hexachloro-cyclohexane, gamma isomer
D014	Methoxychlor	EP Toxic	1,1,1-Trichloro-2,2-bis[p-methoxyphenyl] ethane
D015	Toxaphene	EP Toxic	$C_{10}H_{10}Cl_8$, Technical chlorinated camphene, 67-69 percent chlorine

Expand on descriptive names, especially K and P wastes. Avoid trade-names

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (if available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
D016	2,4-D	EP Toxic	2,4-D (2,4-Dichlorophenoxy-acetic acid)
D017	2,4,5-TP Silvex	EP Toxic	2,4,5-Trichlorophenoxypropionic acid
F001	Spent halogenated solvents used in degreasing	Toxic	Tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, carbon tetrachloride, chlorinated fluorocarbons
F002	Spent halogenated solvents	Toxic	Tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, chlorobenzene, 1,1,2-trichloro-1,2,2, trifluoroethane, ortho-dichlorobenzene, trich-lorofluoromethane
F003	Spent non-halogenated solvents	Ignitable	N.A.
F004	Spent non-halogenated solvents	Toxic	Cresols and cresylic acid, nitrobenzene
F005	Spent non-halogenated solvents	Ignitable, Toxic	Toluene, methyl ethyl ketone, carbon disulfide, isobutanol, pyridine
F006	Wastewater treatment sludges from electro-	Toxic	Cadmium, hexavalent chromium, nickel, cyanide (complexed)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (if available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
F019	Wastewater treatment sludges	Toxic	Hexavalent chromium, cyanide (complexed)
K002	Wastewater treatment sludge	Toxic	Hexavalent chromium, lead
K003	Wastewater treatment sludge	Toxic	Hexavalent chromium, lead
K004	Wastewater treatment sludge	Toxic	Hexavalent chromium
K005	Wastewater treatment sludge	Toxic	Hexavalent chromium, lead
K006	Wastewater treatment sludge	Toxic	Hexavalent chromium
K007	Wastewater treatment sludge	Toxic	Cyanide (complexed), hexavalent
K094	Distillation bottoms	Toxic	Phthalic anhydride
K025	Distillation bottoms	Toxic	Meta-dinitrobenzene, 2,4-dinitrotoluene
K026	Stripping still tails	Toxic	Paraldehyde, pyridines, 2-picoline

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (if available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
K008	Oven residue	Toxic	Hexavalent chromium
K009	Distillation bottoms	Toxic	Chloroform, formaldehyde, methylene chloride, methyl
K010	Distillation side cuts	Toxic	Chloroform, formaldehyde, methylene chloride, methyl chloride, paraldehyde, formic acid, chloroacetaldehyde
K014	Bottoms from acetonitrile purification	Toxic	Acetonitrile, acrylamide
K015	Still bottoms from distillation	Toxic	Benzyl chloride, chlorobenzene, toluene, benzotrichloride
K016	Heavy ends or distillation residues	Toxic	Hexachlorobenzene, hexachlorobutadiene, carbon tetrachloride, hexachloroethane, perchloroethylene
K017	Heavy ends (still bottoms)	Toxic	Epichlorohydrin, chloroethers (bis(chloromethyl) ether and bis(2-chloroethyl) ethers), trichloropropane, dichloropropanols
K018	Heavy ends	Toxic	1,2-dichloroethane, trichloroethylene, hexachlorobutadiene, hexachlorobenzene

TABLE C (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (If available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
K019	Heavy ends	Toxic	Ethylene dichloride, 1,1,1-trichloroethane, 1,1,2-trichloroethane, and 1,1,1,2-tetrachloroethane, trichloroethylene, tetrachloroethylene, carbon tetrachloride, chloroform, vinyl chloride, vinylidene chloride
K020	Heavy ends	Toxic	Ethylene dichloride, 1,1,1-trichloroethane, 1,1,2-trichloroethane, tetrachloroethanes (1,1,2,2-tetrachloroethane and 1,1,1,2-tetrachloroethane), trichloroethylene, tetrachloroethylene, carbon tetrachloride, chloroform, vinyl chloride, vinylidene chloride
K021	Aqueous spent antimony catalyst	Toxic	Antimony, carbon tetrachloride, chloroform
K022	Distillation bottom tars	Toxic	Phenol, tars (polycyclic aromatic hydrocarbons)
K023	Distillation light ends	Toxic	Phthalic anhydride, maleic anhydride
K024	Distillation bottoms	Toxic	Phthalic anhydride, 1,4-naphthoquinone
K093	Distillation light ends	Toxic	Phthalic anhydride, maleic anhydride

TABLE C-4 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (if available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
K028	Spent catalyst	Toxic	1,1,1-trichloroethane, vinyl chloride
K029	Product steam stripper	Toxic	1,2-dichloroethane, 1,1,1-trichloroethane, vinyl chloride, vinylidene chloride, chloroform
K095	Distillation bottoms	Toxic	1,1,2-trichloroethane, 1,1,1,2-tetrachloroethane, 1,1,2,2-tetrachloroethane
K096	Heavy ends	Toxic	1,2 dichloroethane, 1,1,1-trichloroethane, 1,1,2-trichloroethane
K030	Column bottoms or heavy ends	Toxic	Hexachlorobenzene, hexachlorobutadiene, hexachloroethane, 1,1,1,2-tetrachloroethane, 1,1,2,2-tetrachloroethane, ethylene dichloride
K105	Separate aqueous stream	Toxic	Benzene, monochlorobenzene, dichlorobenzenes, 2,4,6-trichlorophenol
K031	By-product salts	Toxic	Arsenic
K048	DAF/float	Toxic	Hexavalent chromium, lead
K049	Slop oil emulsion solids	Toxic	Hexavalent chromium, lead
K050	Heat exchanger cleaning sludge	Toxic	Hexavalent chromium
K051	API separator sludge	Toxic	Hexavalent chromium, lead
K052	Tank bottoms	Toxic	Lead

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (If available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
K061	Emission control dust/sludge	Toxic	Hexavalent chromium, lead, cadmium
K062	Spent pickle liquor	Corrosive, Toxic	Hexavalent chromium, lead
K069	Emission control dust/sludge	Toxic	Hexavalent chromium, lead, cadmium
K100	Waste leaching solution	Toxic	Hexavalent chromium, lead, cadmium
K084	Wastewater treatment sludges	Toxic	Arsenic
K101	Distillation tar residues	Toxic	Arsenic
K102	Residue from activated carbon	Toxic	Arsenic
K071	Brine purification muds	Toxic	Mercury
K106	Wastewater treatment sludge	Toxic	Mercury
K073	Chlorinated hydrocarbon wastes	Toxic	Chloroform, carbon tetrachloride, hexachloroethane, trichloroethane, tetrachloroethylene, dichloroethylene 1,1,2,2-tetrachloroethane
K083	Distillation bottoms	Toxic	Aniline, diphenylamine, nitrobenzene, phenylenediamine

TABLE C (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (if available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
K103	Process residues	Toxic	Aniline, nitrobenzene, phenylenediamine
K104	Combined wastewater streams	Toxic	Aniline, benzene, diphenylamine, nitrobenzene, phenylenediamine
K085	Distillation or fractionation column bottoms	Toxic	Benzene, dichlorobenzenes, trichlorobenzenes, tetrachlorobenzenes, pentachlorobenzene, hexachlorobenzene, benzyl chloride
K105	Separated aqueous stream	Toxic	Benzene, monochlorobenzene, dichlorobenzenes, 2,4,6-trichlorophenol
K032	Wastewater treatment sludge	Toxic	Hexachlorocyclopentadiene
K033	Wastewater and scrub water	Toxic	Hexachlorocyclopentadiene
K034	Filter solids	Toxic	Hexachlorocyclopentadiene
K097	Vacuum stripper discharge	Toxic	Chlordane, heptachlor

TA[illegible] C-[illegible]ont[illegible]d)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA IDENTIFICATION NUMBER (If available)	WASTE COMMON NAME	HAZARD CHARACTERISTIC (i.e., corrosive, toxic ignitable)	BASIS FOR HAZARD DESIGNATION (EPA number, flash point, pH, or EP toxicity constituents and concentrations)
K035	Wastewater treatment sludges	Toxic	Creosote, chrysene, naphthalene, fluoranthene benzo(b) fluor-anthene, benzo(a)pyrene, indeno (1,2,3-cd) pyrene, benzo(a)anthracene, dibenzo (a)anthracene, acenaphthalene
K036	Still bottoms	Toxic	Toluene, phosphorodithoic and phosphorothioic acid esters
K037	Wastewater treatment sludges	Toxic	Toluene, phosphorodithioic and phosphorothioic acid esters
K038	Wastewater from washing and stripping	Toxic	Phorate, formaldehyde, phosphorodithioic and phosphorothioic acid esters
K039	Filter cake	Toxic	Phosphorodithioic and phosphorothioic acid esters
K040	Wastewater treatment sludge	Toxic	Phorate, formaldehyde, phosphorodithioic and phosphorothioic acid esters
K041	Wastewater treatment sludge	Toxic	Toxaphene

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

The following list of materials is considered to be toxic or acutely toxic. The basis for listing is inherent with the individual compound. Another known basis or multiple bases are indicated by capital letters in parentheses: R = reactive; I = ignitable; C = corrosive; T = toxic. If no letter is shown, the compound should be considered as singularly acutely toxic.

EPA HAZARDOUS WASTE NO.	SUBSTANCE
P001	3-(alpha-acetonylbenzyl)-4-hydroxycoumarin and salts
P002	1-Acetyl-2-thiourea
P003	Acrolein, Agarin (see P007), Agrosan GN 5 (see P092), Aldicarb (see P069), Aldifen (see P048)
P004	Aldrin, Algimycin (see P092)
P005	Allyl alcohol
P007	Aminoethylene (see P054), 5-(Aminomethyl)-3-isoxazolol
P008	4-Aminopyridine, Ammonium metavanadate (see P119)
P010	Arsenic acid
P011	Arsenic pentoxide
P012	Arsenic trioxide, Athrombin (see P001), AVITROL (see P009), Aziridene (see P054), AZOGOD (see P061), Azophos (see P061), BANTU (see P072)
P014	Benzenethiol, Benzoepin (see P050)
P015	Beryllium dust
P016	Bis(chloromethyl) ether, BLADAN-M (see P071)
P017	Bromoacetone
P020	2-sec-Butyl-4,6-dinitrophenol
P023	Chloroacetaldehyde
P024	p-Chloroaniline
P025	1-(p-Chlorobenzoyl)-5-methoxy-2-methylindole 3-acetic acid
P026	1-(o-Chlorophenyl) thiourea

Describe the waste containing these constituents (sludges, filter cake)

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
P027	3-Chloropropiontrile
P028	Alpha-Chlorotoluene
P034	2-Cyclohexyl-4,6-dinitrophenol, D-CON (see P001), DETHMOR (see P001), DETHNEL (see P001), DFP (see P043)
P036	Dichlorophenylarsine, Dicyanogen (see P031)
P037	Dieldrin, DIELDREX (see P037)
P038	Diethylarsine
P040	0,0-Diethyl-0-(2-pyrazinyl) phosphorothioate
P041	0,0-Diethyl phosphoric acid, 0-p-nitropheynyl ester
P042	3,4-Dihydroxy-alpha-(methylamino)-methyl benzyl alcohol
P043	Di-isopropyl fluorophosphate, DIMETATE (see P044), 1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexa-chloro-1,4,4a,5,8,8a-hexahydro endo, endo (see P060)
P044	Dimethoate
P045	3,3-Dimethyl-1-(methylthio), 2-butanone-0-[methylamino carbonyl] oxime
P048	2,4-Dinitrophenol, DINOSEB (see P020), DINOSEBE (see P020), Disulfoton (see P039)
P049	2,4-Dithiobiuret, DNBP (see P020), DOLCO MOUSE CEREAL (see P108), DOW GENERAL (see P020), DOW GENERAL WEED KILLER (see P020), DOW SELECTIVE WEED KILLER (see P020), DOWICIDE G (see P090), DYANACIDE (see 092), EASTERN STATES DUOCIDE (see P001), ELGETOL (see P020)
P051	Endrin, Epinephrine (see P042)
P054	Ethylenimine, EASCO FASCRAT POWDER (see P001), FEMMA (see P091)

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
P056	Fluorine
P057	2-Fluoroacetamide
P058	Fluoroacetic acid, sodium salt, FOLODOL-80 (see P071), FOLODOL M (see P071), FOSFERNO M 50 (see P071), FRATOL (see P058), Fulminate of mercury (see P065), FUNGITOX OR (see P092), FUSSOF (see P057), GALLOTOX (see P092), GEARPHOS (see P071), GERUTOX (see P020)
P066	Methomyl
P067	2-Methylaziridine, METHYL-E 605 (see P071)
P068	Methyl hydrazine, Methyl isocyanate (see P064)
P069	2-Methyllactonitrile
P070	2-Methyl-2-(methylthio)propionaldehyde-o-(methylcarbonyl) oxime, METHL NIRON (see P042)
P072	1-Naphthyl-2-thiourea
P073	Nickel carbonyl
P075	Nicotine and salts
P076	Nitric oxide
P077	p-Nitroaniline
P078	Nitrogen dioxide
P087	Osmium tetroxide
P089	Parathion, PCP (see P090), PENNCAP-M (see P071), PENOXYL CARBON N (see P048)
P094	Phorate
P101	Propanenitrile

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
P102	2-Propyn-1-ol PROTHROMADIN (see P001), QUICKSAM (see P092), QUINTOX (see P037), RAT AND MICE BAIT (see P100), RAT-A-WAY (see P001), RAT-B-GON (see P001), RAT-O-CIDE #2 (see P001), RAT GUARD (see P001), RAT-KILL (see P001), RAT-MIX (see P001), RATS-NO-MORE (see P001), RAT-OLA (see P001), RATOREX (see P001), RATTUNAL (see P001), RAT-TROL (see P001), RO-DETH (see P001), RO-DEX (see P108), ROSEX (see P001), ROUGH and READY MOUSE MIX (see P001), SANASEED (see P108), SANTOBRITE (see P090), SANTOPHEN (see P090), SANTOPHEN 20 (see P090), SCHRADAN (see P085)
P103	Selenourea
P107	Strontium sulfide
P108	Strychnine and salts, SUBTEX (see P020), SYSTAM (see P085), TAG FUNGICIDE (see P092), TEKWAISA (see P0710, TEMIC (see P070), TEMIK (see P070), TERM-I-TROL (see P090)
P109	Tetraethyldithiopyrophosphate
P110	Tetraethyl lead
P111	Tetraethylpyrophosphate
P115	Thallium (1) sulfate, THIFOR (see P092), THIMUL (see P092), THIODAN (see P050), THIOFOR (see P050), THIOMUL (see P050), THIONEX (see P050), THIOPHENIT (see P071)
P118	Trichloromethanethiol, TWIN LIGHT RAT AWAY (see P001), USAF RH-8 (see P069), USAF-EK-4890 (see P002)
P119	Vanadic acid, ammonium salt
P123	Toxaphene
U001	Acetaldehyde (I)
U002	Acetone (I)
U003	Acetonitrile (I,T)
U004	Acetophenone
U005	2-Acetylaminoflourene

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U007	Acrylamide, Acetylene tetrachloride (see U209), Acetylene trichloride (see U228)
U008	Acrylic acid (1)
U009	Acrylonitrile, AEROTHENE TT (see U226), 3-Amino-5-(p-acetamidophenyl) 1H-1,2,4-triazole, hydrate (see U011)
U010	6-Amino-1,1a,2,8,8a,8b-hexahydro-8-(hydroxymethyl) 8-methoxy-5-methylcarbamate azirino (2,3,3,4) pyrrolo (1,2-a) indole-4,-7-dione (ester)
U011	Amitrole
U012	Aniline (i)
U014	Auramine
U015	Azaserine
U016	Benz[c]acridine
U017	Benzal chloride
U018	Benz[a]anthracene
U019	Benzene
U021	Benzidine, 1,2-Benzisothiazolin-3-one, 1,1-dioxide (see U202), Benzo [a] anthracene (see U018)
U022	Benzo[a]pyrene
U024	Bis(2-chloroethoxy) methane
U025	Bis(2-chloroethyl) ether
U026	N,N-Bis(2-chlorothyl)-2-napthylamine
U027	Bis(2-chloroisopropyl) ether
U028	Bis(2-ethylhexyl) phthalate
U029	Bromomethane
U030	4-Bromophenyl phenyl ether
U031	n-Butyl alcohol (I)

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U032	Calcium chromate, Carbolic acid (see U1880, Carbon tetrachloride (see U211)
U033	Carbonyl fluoride
U034	Chloral
U035	Chlorambucil
U036	Chlordane
U037	Chlorobenzene
U038	Chlorobenzilate
U039	p-Chloro-m-cresol
U040	Chlorodibromomethane
U041	1-Chloro-2,3-epoxypropane, CHLOROETHENE NU (see U226)
U042	2-Chloroethyl vinyl ether
U043	Chloroethene
U044	Chloroform (I,T)
U045	Chloromethane (I,T)
U046	Chloromethyl methyl ether
U047	2-Chloronaphthalene
U048	2-Chlorophenol
U049	4-Chloro-o-toluidine, hydrochloride
U050	Chrysene, C.I. 23060 (see U073)
U051	Creosote
U052	Cresols
U053	Crotonaldehyde
U054	Cresylic acid

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U055	Cumene, Cyanomethane (see U003)
U056	Cyclohexane (I)
U057	Cyclohexanone (I)
U058	Cyclophosphamide
U059	Daunomycin
U060	DDD
U061	DDT
U062	Diallate
U063	Dibenz[a,h]anthracene, Dibenzo[a,h]anthracene (see U063)
U064	Dibenzo[a.i]pyrene
U065	Dibromochloromethane
U066	1,2-Dibromo-3-chloropropane
U067	1,2-Dibromoethane
U068	Dibromomethane
U069	Di-n-butyl phthalate
U070	1,2-Dichlorobenzene
U071	1,3-Dichlorobenzene
U072	1,4-Dichlorobenzene
U073	3,3-Dichlorobenzidine
U074	1,4-Dichloro-2-butene (I,T), 3,3-Dichloro-4,4, diaminobiphenyl (see U073)
U075	Dichlorodifluoromethane
U076	1,1-Dichloroethane
U077	1,2-Dichloroethane

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U078	1,1-Dichloroethylene
U079	1,2-trans-dichloroethylene
U080	Dichloromethane, Dichloromethylbenzene (see U017)
U081	2,4-Dichlorophenol
U082	2,6-Dichlorophenol
U083	1,2-Dichloropropane
U084	1,3-Dichloropropane
U085	Diepoxybutane (I,T)
U086	1,2 Diethylhydrazine
U087	0,0-Diethyl-S-methyl ester of phosphorodithioic acid
U088	Diethyl phthalate
U089	Diethylstilbestrol
U090	Dihydrosafrole
U091	3,3-Dimethylbenzidine
U092	Dimethylamine (I)
U093	p-Dimethylaminoazobenzene
U094	7,12-Dimethylbenz[a]anthracene
U095	3,3-Dimethylbenzidine
U097	Dimethylcarbamoyl chloride
U098	1,1-Dimethylhydrazine
U099	1,2-Dimethylhydrazine
U100	Dimethylnitrosoamine

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U101	2,4-Dimethylphenol
U102	Dimethyl phthalate
U103	Dimethyl sulfate
U104	2,4-Dinitrophenol
U105	2,4-Dinitrotoluene
U106	2,6-Dinitrotoluene
U107	Di-n-octyl phthalate
U108	1,4-Dioxane
U109	1,2-Diphenylhydrazine
U110	Dipropylamine (I)
U111	Di-N-propylnitrosamine, EBDC (see U114), 1,4-Epoxybutane (see U213)
U112	Ethyl acetate (I)
U113	Ethyl acrylate (I)
U114	Ethylenebisdithiocarbamate
U115	Ethylene oxide (I,T)
U116	Ethylene thiourea
U117	Ethyl ether (I,T)
U118	Ethylmethacrylate
U119	Ethyl methanesulfonate, Ethyinitrile (see U003), Firemaster and 23P (see U235)
U120	Fluoranthene
U121	Fluorotrichloromethane
U122	Formaldehyde
U123	Formic acid (C,T)

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U124	Furan (I)
U125	Furfural (I)
U126	Glycidyladehyde
U127	Hexachlorobenzene
U128	Hexachlorobutadiene
U129	Hexachlorocyclohexane
U130	Hexachlorocyclopentadiene
U131	Hexachloroethane
U132	Hexachlorophene
U134	Hydrofluoric acid (C,T)
U135	Hydrogen sulfide, Hydroxybenzene (see U188)
U136	Hydroxydimethylarsine oxide, 4,4-(Imidocarbonyl) bis(N,N-dimethyl)aniline (see U014)
U137	Indeno(1,2,3,-cd)pyrene
U138	Iodomethane
U139	Iron dextran
U140	Isobutyl alcohol (I,T)
U141	Isosafrole
U142	Kepone
U143	Lasiocarpine
U144	Lead acetate
U145	Lead phosphate
U146	Lead subacetate
U147	Maleic anhydride

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U171	2-Nitropropane (I)
U172	N-Nitrosodi-n-butylamine
U173	N-Nitrosodiethanolamine
U174	N-Nitrosodiethylamine
U175	N-Nitroso-N-propylamine
U176	N-Nitroso-N-ethylurea
U177	N-nitro-N-methylurea
U178	N-Nitroso-N-methylurethane
U179	N-Nitrosopiperidine
U180	N-Nitrosopyrrolidine
U181	5-Nitro-o-toluidine
U182	Paraldehyde, PCNB (see U185)
U183	Pentachlorobenzene
U184	Pentachloroethane
U185	Pentachloronitrobenzene
U186	1,3-Pentadiene(I), Perc(see U210), Perchlorethylene (see U210)
U187	Phenacetin
U188	Phenol
U190	Phthalic anhydride
U191	2-Picoline
U192	Pronamide
U193	1,3-Propane sultone
U194	n-Propylamine (I,T)

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U196	Pyridine
U197	Quinones
U200	Reserpine
U201	Resorcinol
U202	Saccharin and salts
U203	Safrole
U204	Selenious acid
U205	Selenium sulfide (R,T), Silvex (see U233)
U206	Streptozótocin, 2,4,5-T (see U232)
U207	1,2,4,5-Tetrachlorobenzene
U208	1,1,1,2-Tetrachloroethane
U209	1,1,2,2-Tetrachloroethane
U210	Tetrachloroethene, Tetrachloroethylene
U211	Tetrachloromethane
U212	2,3,4,6-Tetrachlorophenol
U213	Tetrahydrofuran (I)
U214	Thallium (I) acetate
U215	Thallium (I) carbonate
U216	Thallium (I) chloride
U217	Thallium (I) nitrate
U218	Thioacetamide
U219	Thiourea
U220	Toluene
U221	Toluenediamine

TABLE C-1 (Continued)

HAZARDOUS MATERIALS MANAGED AT PROTECO FACILITY

EPA HAZARDOUS WASTE NO.	SUBSTANCE
U222	o-Toluidine hydrochloride
U224	Toxaphene, 2,4,5,=TP (see U233)
U225	Tribromomethane
U226	1,1,1-Trichloroethane
U227	1,1,2-Trichloroethane
U228	Trichloroethene, Trichloroethylene (see U228)
U229	Trichlorofluoromethane
U230	2,4,5-Trichlorophenol
U231	2,4,6-Trichlorophenol
U232	2,4,5-Trichlorophenoxyacetic acid
U233	2,4,5-Trichlorophenoxypropionic acid alpha, alpha, alpha-Trichlorotoluene (see U023), TRI-CLENE (see U228)
U235	Tris(2,3-dibromopropyl) phosphate
U236	Trypan blue
U237	Uracil mustard
U238	Urethane, Vinyl chloride (see U043), Vinylidene chloride (see U078)
U239	Xylene (I)
U240	2,44 D, salt and esters
U242	Pentachlorophenol
U243	Hexachloropropene
U244	Thiram
U247	Methoxychlor

The percentage of wastes which will be managed at the PROTECO site will vary over time, depending on: 1) the pace and scope of remedial cleanup and closure programs -- both governmental and private; 2) the nature and type of industrialization on the island; and 3) future regulatory changes and resultant changes in waste management practices.

The above lists of wastes are based on th best current judgment of future industrial and regulatory trends. Because of these factors, it is not possible to definatively state exact waste percentages, particularly over the life of the facility. As this Part B Application is for proposed new facilities, the actual mix of wastes managed will depend on the market as it develops. PROTECO will carefully review all candidate waste streams, however, and only accept wastes which can be properly and safely managed as per this Waste Analysis Plan.

The stabilization/fixation treatment procedure renders a waste to a material containing no free liquids by the reaction of a cement kiln dust, water and waste. This material is then disposed of in the secure landfill. In general, waste mixtures to be stabilized will have a maximum organic concentration of one percent.

Hazardous wastes are stored at the PROTECO facility in the container storage facility in sealed containers which are compatible with the wastes. The drums holding hazardous wastes will be stored in separate sections within the container storage area due to general and specific incompatibilities of the wastes. This area is discussed in greater detail in Section D.

The proposed tank farm will contain eight tanks for storage and/or treatment of wastes before additional treatment and/or disposal. The capacity and waste types for each tank are shown in Drawing B511-D-P20.

C-1b <u>General Description of Hazardous Wastes Treated, Stored or Disposed of at the Facility</u>

PROTECO treats, stores and disposes of a broad range of hazardous wastes including solids, slurries, sludges and liquids. The wastes stored

at the facility can be generally grouped in the following categories as defined in 40 CFR Part 261:

Ignitable
Corrosive
Extraction Procedure (EP) Toxic
Toxic
Acutely Hazardous

Some of the hazardous wastes that will be accepted for treatment, storage or disposal at the facility are summarized in Table C-2, and include the following:

- Ignitable Mixtures (D001) - classified as hazardous due to their flammable characteristic. Wastes in this group have a flash point less than 140°F (60°C). The solvents accepted in this group include acetone, methanol, ethanol and isopropyl alcohol. In addition various oils, paint wastes, liquid tars, lacquer chip and lacquer base that are also flammable are accepted at the facility.

- Acidic Solutions (D002) - classified as hazardous due to their corrosive characteristics. All wastes in this category have a pH less than or equal to 2. The typical liquids accepted in this group includes acid cleaning solutions, ferric chloride solution, sulfuric acid and hydrochloric acid.

- Alkaline (Caustic) Solutions (D002) - classified as hazardous due to their corrosive characteristics. All wastes in this category have a pH greater than or equal to 12.5. This group typically includes alkaline cleaning solutions, sodium hydroxide and other wastes generated in the production of Chlorox.

- EP Toxic Wastes - these are metal bearing wastes that are not listed in 40 CFR Part 261. These wastes are classified as hazardous due to EP Toxic concentrations of metals greater than or

TABLE C-2 (CONTINUED)

SOME HAZARDOUS WASTES ACCEPTED AT PROTECO

EPA Hazardous Waste No	Waste Classification	Wastes Included
P105	Commercial Chemical Products	Off-spec chemicals
U002, U112	Commercial Chemical Products	Ignitable off-spec chemical products
U154, U239	Commercial Chemical Products	
U019, U220	Commercial Chemical Products	Ignitable and toxic off-spec chemical products.

Hazardous Wastes
Not Accepted at PROTECO

Shock Sensitive Waste
Radioactive Wastes
Highly Reactive Wastes
Explosives
Pressurized Gases in Cylinders
TSCA - Regulated Wastes
Pyrophoric Wastes
F020
F021
F022
F023
F026
F027

equal to the values listed in Table C-3. These wastes typically are generated from the production of hair and beauty products, electroplating operations and the manufacture of electrical products.

- Spent Halogenated Solvents (F001, F002) - listed as hazardous wastes due to their toxic characteristics. Examples of this waste that are accepted at the site include methylene chloride, 1,1,1-trichloroethane, tetrachloroethylene and the still bottoms generated from their use in manufacturing operations. These wastes are generated primarily from the cleaning operations associated with the production of pharmaceutical products and the manufacturing of electrical components.

- Spent Non-Halogenated Solvents (F003, F005) - listed as hazardous wastes due to their ignitable and/or toxic characteristics. Examples of this waste accepted at the site include acetone, ethyl acetate, xylene, and toluene. These wastes are typically generated in Puerto Rico from the production of pharmaceutical products and diagnostic medical devices.

- Wastewater Sludges from Electroplating Operations (F006) - listed as hazardous due to their toxic properties. The hazardous constituents for which this waste is listed typically include cadmium, hexavalent chromium or nickel. These wastes are generated by various electroplating operations in Puerto Rico.

- Pesticide Waste (D013) - classified as hazardous due to an EP Toxic concentration of lindane greater than 0.4 ppm. This waste is generated in Puerto Rico from a shampoo manufacturing operating process.

- Leaded Tank Bottoms from the Petroleum Refining Industry (K052) - listed as a hazardous waste due to its toxicity and hazardous concentrations of lead. This waste is generated during the refining and storage of petroleum.

TABLE C-3

MINIMUM CONCENTRATION OF METALS FOR WASTES TO BE CLASSIFIED AS "EP TOXIC"

EPA Number	Contaminant	Minimum Concentration (milligrams per liter)
D004	Arsenic	5.0
D006	Cadmium	1.0
D007	Chromium	5.0
D008	Lead	5.0
D009	Mercury	0.2
D010	Selenium	1.0
D011	Silver	5.0
D013	Lindane	0.4

The six (6) major waste streams which will be processed at the Proteco site are waste lindane solution (D013), corrosive wastes (D002), ignitable wastes (D001), lead bearing wastes (D008) and spent halogenated wastes (F001, F002), and CERCLA (Superfund) site cleanup wastes.

Table C-2 also includes a listing of those types of wastes not accepted at the PROTECO facility.

C-2 Initial Waste Characterization [40 CFR 270.14(b)(2)]

C-2a Proposed Waste Characterization Program

C-2a(1) Waste Pre-Acceptance Sheet. Proteccion Tecnica Ecologica, Inc., accepts both hazardous and non-hazardous waste streams for storage, treatment and/or disposal from many waste generators (private and governmental) in the Commonwealth of Puerto Rico. Since it is necessary for PROTECO to characterize a hazardous waste to determine if it can be successfully treated, stored or disposed of, prior to the acceptance of any hazardous wastes, a prospective client company (i.e., any generator including small quantity generators) will be required to fully document the characteristics of each hazardous waste stream to be stored, treated and/or disposed of at the facility. This information is recorded by the prospective client on a PROTECO Waste Pre-acceptance Sheet (WPS) provided by PROTECO for this purpose. A copy of the WPS is shown in Figure C-1.

The WPS requires a generator to provide information on the process generating the waste, the physical properties of the waste and the chemical properties of the waste including chemical components and concentrations of heavy metals. In addition, PROTECO requires submittal of a copy of all laboratory analyses performed by the generator to determine the hazardous nature of the waste. PROTECO also requires submittal of material safety data sheets (MSDS) from the generator to augment the WPS whenever they are applicable and available.

All of the waste characterization sheets detailing physical and chemical properties of the hazardous wastes accepted by PROTECO are contained

FIGURE C-1

Waste Pre-Acceptance Sheet

A. GENERAL INFORMATION

GENERATOR NAME _____ TRANSPORTER _____

FACILITY ADDRESS _____ TRANSPORTER PHONE _____

_____ GENERATOR USEPA ID _____

_____ GENERATOR STATE ID _____

TECHNICAL CONTACT _____ TITLE _____ PHONE _____

NAME OF WASTE _____

PROCESS GENERATING WASTE _____

B. PHYSICAL CHARACTERISTICS OF WASTE

COLOR	ODOR	PHYSICAL STATE @ 70°F	LAYERS	FREE LIQUIDS	
	__ NONE __ MILD				
	__ STRONG	__ SOLID __ SEMI-SOLID	__ MULTILAYERED	__	__
DESCRIBE _____		__ LIQUID __ POWDER	__ BI-LAYERED	__ YES	__ NO
	__ SINGLE PHASED	VOLUME _____			

PH			SPECIFIC GRAVITY			FLASH POINT	
__ .2	__ 7.1-10.0	__ N/A	__ .8	__ 1.3-1.4	__ 70°F	__ 200°F	__ CLOSED CUP
__ 2-4	__ 10.1-12.5		__ .8-1.0	__ 1.5-1.7	__ 70°F-100°F	__ NO FLASH	__ OPEN CUP
__ 4.1-6.9	__ 12.5		__ 1.1-1.2	__ 1.7	__ 101°F-130°F	__ EXACT ____	
__ 7	__ EXACT ____		__ EXACT ____		__ 140°F-200°F		

C. CHEMICAL COMPOSITION (Totals must add to 100%)

_____ _____ %
_____ _____ %
_____ _____ %
_____ _____ %
_____ _____ %
_____ _____ %
_____ _____ %
_____ _____ %
_____ _____ %
_____ _____ %
_____ _____ %

D. METALS __ TOTAL (PPM) __ EPA EXTRACTION PROCEDURE (mg)

ARSENIC	_____	SELENIUM	_____
BARIUM	_____	SILVER	_____
CADMIUM	_____	COPPER	_____
CHROMIUM	_____	NICKEL	_____
MERCURY	_____	ZINC	_____
LEAD	_____	THALLIUM	_____
CHROMIUM (Hexavalent)	_____	_____	_____

E. OTHER COMPONENTS TOTAL (PPM)

CYANIDES _____ PCB's _____

SULFIDES _____ PHENOLICS _____

(0059c)

FIGURE C-1 (CONTINUED)

Waste Pre-Acceptance Sheet (CONTINUED)

F. SHIPPING INFORMATION

DOT HAZARDOUS MATERIAL __ YES __ NO

PROPER SHIPPING NAME _____

HAZARD CLASS _____ ID NO _____ RO _____

METHOD OF SHIPMENT __ BULK LIQUID __ BULK SOLID

__ DRUM (TYPE/SIZE) _____

ANTICIPATED VOLUME _____ GALS _____ CUBIC YARDS

_____ OTHER _____

PER __ ONE TIME __ WEEK __ MONTH

__ QUARTER __ YEAR __ _____

G. HAZARDOUS CHARACTERISTICS

REACTIVITY __ NONE __ PYROPHORIC __ SHOCK SENSITIVE

__ EXPLOSIVE __ WATER REACTIVE __ OTHER _____

OTHER HAZARDOUS CHARACTERISTICS

__ NONE __ RADIOACTIVE __ ETIOLOGICAL

__ PESTICIDE MANUFACTURING WASTE __ OTHER _____

USEPA HAZARDOUS WASTE __ YES __ NO

USEPA HAZARDOUS CODES _____ _____ _____ _____

STATE HAZARDOUS WASTE __ YES __ NO

STATE CODES _____

H. SPECIAL HANDLING INFORMATION _____

I HEREBY CERTIFY THAT ALL INFORMATION SUBMITTED ON THIS AND ALL ATTACHED DOCUMENTS IS COMPLETE AND ACCURATE AND THAT ALL KNOWN OR SUSPECTED HAZARDS HAVE BEEN DISCLOSED.

AUTHORIZED SIGNATURE _____ TITLE _____ DATE _____

in a bound file at the laboratory office. These sheets are kept as part of the operating record as required by 40 CFR Part 264.74 and will be kept for the life of the facility.

After PROTECO finishes the construction and outfitting their own laboratory, by late 1986, PROTECO will begin to require all current generators to send PROTECO a representative sample of the waste described in the WPS. All new generators will be required to submit both an updated WPS and a representative sample.

When a representative sample or new WPS and representative sample are returned to PROTECO, the PROTECO laboratory chemist will review the sheet for content and completeness and the representative sample will be analyzed by PROTECO as described in the Waste Analysis Plan (Section C-3). If the WPS returned to PROTECO is incomplete, the PROTECO chemist will telephone to request additional information from the generator. This information will be added to the generator's WPS by the PROTECO chemist. PROTECO will decline to accept the waste if a generator refuses to provide PROTECO with a WPS and a representative sample of the waste.

When PROTECO has determined that the WPS is complete, a review will commence to determine if the waste can be handled by the facility in an environmentally safe manner. Based on the following, PROTECO will decide whether to initially accept the waste.

- PROTECO operating procedures and available capacities
- Waste streams presently treated, stored or disposed by PROTECO
- Waste characterization sheet
- Generator's laboratory analysis of the waste
- Material Safety Data Sheets
- PROTECO's analysis of the representative sample
- Ability of PROTECO's facilities to manage the waste.

In addition to keeping all waste characterization sheets, PROTECO will keep all laboratory reports detailing physical and chemical properties of the hazardous wastes accepted by PROTECO in a bound file at the site

laboratory. These reports will be kept as part of the operating record as required by 40 CFR Part 264.74 and will be kept for the life of the facility.

C-2a(2) Generator Audits. As of August 1985, the PROTECO chemist, salesman or other authorized representative of the facility visited major generators to conduct a "generator audit". This audit was conducted in addition to requesting a WPS and a representative sample from the generator. The audit allowed PROTECO to obtain a copy of the generator's process flow sheets, review the generator's processes that produce hazardous waste, determine the generator's knowledge about these wastes and see the generator's storage area and the containers used to transport the waste. A total of twelve facilities were audited as the initial phase of this program. From these audits, PROTECO has identified the following:

- Major constituents in the waste and their percent ranges
- Types of cross contamination that could occur
- Generator's procedures to avoid contamination
- Errors or inconsistencies that could occur during shipping
- Waste to container compatibility
- Estimated annual production of the waste
- All known waste characterization data and its sources

A copy of the Generator Audit Form used by PROTECO in conducting these audits is provided in Figure C-2. This form will be kept in the generator's file at the laboratory office for the life of the facility.

As each generator audit is conducted by PROTECO, it will be noted in the generator audit log. The log will be kept as part of the facility operating record. Once a year, the log will be reviewed by the management staff of PROTECO to determine if any generators should be reaudited. A copy of the generator audit log that will be used is provided in Figure C-3.

FIGURE C-2

PROTECO

GENERATOR AUDIT FORM

GENERATOR _____

GENERATOR EPA ID # _____

LOCATION _____
_____

TELEPHONE _____

CONTACT PERSONS _____
_____

SIC CODE FOR PRINCIPLE PRODUCTS (4 DIGIT NO.)

a. _____ b. _____ c. _____

HAZARDOUS WASTE GENERATED AT THE FACILITY

	Waste Description	EPA ID #	Quantity Generated/Month
a.			
b.			
c.			
d.			
e.			
f.			

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FIGURE C-2 (CONTINUED)

PART II WASTE DATA

NAME OF WASTE _____

PROCESS PRODUCING WASTE _____

_____

POTENTIAL AND TYPES OF CONTAMINATION _____

_____

_____

STORAGE CONTAINER _____

PHYSICAL INFORMATION

LIQUID _____ SOLID _____ SLUDGE _____ SLURRY _____

SP. GRAVITY _____ pH _____ FLASH POINT _____

COLOR _____ ODOR _____ TEXTURE _____

CHEMICAL INFORMATION

AVERAGE COMPONENT	CONCENTRATION %	AVERAGE HEAVY METALS	CONCENTRATION PPM
a.			
b.			
c.			
d.			
e.			

Sulfur _____ Chlorine _____

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FIGURE C-3

GENERATOR AUDIT LOG

Date	Generator	Address	Contact	Telephone No.	Auditor

C-2a(3) Generator/PROTECO Agreements. Once PROTECO agrees to accept a waste, the generator must agree to inform PROTECO of any changes in the hazardous waste received by the facility due to changes in raw materials, generator's processes or operational changes. The generator and PROTECO will also determine the variability that will be allowed to occur in the generator's waste. Waste variability will be determined by means of the WPS, analysis by PROTECO of the representative sample and the generator audit. For example, a waste may contain 20-30 percent methanol, 70 percent isopropyl alcohol and up to 10 percent water. PROTECO will determine if these ranges are acceptable to the facility. Again, the generator must agree to notify PROTECO of any changes in the variability of the wastes.

C-3 Waste Analysis Plan
[40 CFR 270.14(b)(3) and 264.13(b)]

C-3a General Waste Parameters and Rationale for their Selection

C-3a(1) Pre-Acceptance Waste Characterization. As discussed in Section C-2, generators will be required to provide PROTECO with an initial pre-acceptance waste characterization. This initial pre-acceptance waste characterization will consist of submitting a completed Waste Pre-acceptance Sheet (WPS), any copies of laboratory analyses conducted on the waste, any Material Safety Data Sheets where applicable and a representative sample of the waste. This will provide PROTECO with the following information:

- Process generating the waste
- Physical properties of the waste
- Chemical properties of the waste including chemical components and their percentages.
- Source of data provided.

PROTECO will analyze each representative sample provided by the generator. The waste parameters analyzed for will be dependent on the classification of the waste and the information provided by the generator on the accompanying WPS.

C-3a(2) Incoming Load Testing Parameters (Fingerprint Parameters). Each incoming load will be tested for standard fingerprint parameters prior to acceptance at PROTECO. These parameters are selected to prevent acceptance of reactive materials, and incorrect storage of incompatible wastes. These parameters also ensure that the incoming waste load conforms to the representative sample sent to PROTECO with the Waste Pre-acceptance Sheet. These fingerprint parameters are listed in Table C-4.

The parameters noted in Table C-4 have been selected on the following basis:

- pH -- This parameter will be tested in ignitable and aqueous wastes, solids and EP Toxic solutions and sludges. For aqueous wastes, this will determine the amount of neutralizing agent required for treatment and will help determine compatibility with the landfill liner system. This will determine if the waste is amenable with stabilization/fixation treatment and to determine compatibility with the landfill liner system.

- Flashpoint -- The flash point will be measured to determine the waste's ignitability and, therefore, special waste handling practices. Some ignitable waste liquids, sludges and solids will be rendered non-ignitable by using stabilization/fixation. Others may be shipped off-site in bulk quantities for incineration. Also, a change in the flash point is indicative of changes in waste concentration ranges.

- Viscosity -- The viscosity will help determine the best method for handling, storing and treating the waste. Changes in viscosity may affect handling, storage and treatment operations; and may indicate change in a waste characteristics or waste concentration ranges.

TABLE C-4

STANDARD INCOMING LOAD
TESTING PARAMETERS FOR ALL WASTES

(Fingerprint Parameters)

pH
Flashpoint
Viscosity and/or Specific Gravity
Free Liquid/Water Content
Cyanides
Sulfides

- Specific Gravity -- The specific gravity is important in determining optimum waste solutions for stabilization/fixation. Specific gravity is also significant because a change in specific gravity is indicative of a change in waste characteristics or waste concentration ranges. Also, density tests can be used as a quick indicator of halogenated organics contamination (such as PCBs) in a waste solvent or oil mixture.

- Free Liquids/Water Content -- The test for free liquids will be used to determine if a waste needs stabilization/fixation prior to landfilling. This test also signifies a change in waste characteristics and waste concentration ratios.

- Cyanide -- This parameter will be used to determine the presence of cyanide in the waste stream. This will help to determine whether the waste should be accepted or rejected since the presence of cyanide can indicate a reactive waste, and reactive wastes are not accepted by the facility.

- Sulfide -- This parameter will be used to determine if sulfide is present in the waste. This will be useful in determining if an aqueous waste, sludge and slurry can be treated by the stabilization/fixation treatment process. It will also be used to determine if a waste will be accepted or rejected since the presence of sulfide can be indicative of a reactive waste, and reactive wastes are not accepted by the facility.

The results of the pre-acceptance analysis of the generator's representative sample will be coded on what will be known as the Standard Reference Form. A copy of the Standard Reference Form is provided in Figure C-4. One copy of the Standard Reference Form stating the results of the analysis performed by the PROTECO laboratory will be kept in the laboratory as the standard laboratory reference. The values that are obtained from the analysis of the incoming load sample will be compared to the values on the Generator's Standard Reference Form. If the values are

FIGURE C-4

PROTECO

STANDARD REFERENCE FORM

Sample No. _____

Generator Name: _____ Date: _____
Address: _____
City: _____ State: _____ Zip Code: _____
Generator Contact: _____ Telephone: _____

Waste to be Analyzed: _____
Process Producing Waste: _____
Date Sampled by the Generator: _____

Processed by: _____
Date Processed: _____

Physical Characteristics

Physical State: _____ Color: _____
Odor: _____ Texture: _____
Specific Gravity: _____ Viscosity: _____

Chemical Characteristics

pH: _____ Flash Point: _____ Water Content: _____
Cyanide: _____ Sulfide: _____ Halides: _____
Oxidizable Organics: _____ Chlorides: _____

Other Applicable Information: _____
_____
_____
_____
_____

Transport Method _____

Container Type _____

Signature: _____

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significantly different, the generator will be contacted to clarify the differences that have been noted.

C-3a(3) Supplemental Analyses.

C-3a(3)a Waste Characterization Testing Parameters. PROTECO analyzes wastes of certain hazard classifications with additional tests. These additional characterizations are specific for each waste and may be adjusted by the PROTECO facility chemist based on information provided by the generator and analytical testing already completed.

The wastes which may be included in this testing program are listed in Table C-5. Discussion of the selection of these additional parameters are given in Table C-6 and Appendix C.1.

C-3a(3)b Process Specific Waste Testing Parameters. Some of the additional testing of wastes at PROTECO are the process specific analyses, shown in Table C-7. The results of these analyses provide the PROTECO chemist with another level of confidence concerning the proper means of treatment, storage and disposal. Some of these additional analyses utilize unique procedures and protocol formulated for PROTECO for proper waste characterization. Others are standard analytical techniques recognized by the USEPA and ASTM.

The rationale behind these process specific tests are given below:

- Oxidizable Organics -- This test will be used to determine easily oxidized organic substances such as aldehydes, alcohols or other simple poly-functional compounds in ignitable compounds and non-halogenated solvents. This will be useful in determining treatment of organic waste by using either stabilization/fixation or resource recovery at an off-site facility.

- Halogen Test -- The halogen test will be used to establish if a waste contains trace or larger quantities of halogens. This information will be useful in determining treatment of a waste by

TABLE C-5

ADDITIONAL WASTE CLASSIFICATION TESTING

Waste Classification	Test Method	Method Source
Halogenated Organics F001 F002	Gas Chromatography	EPA Method 8010 (Halogenated Volatile Organics)
Cadmium Contaminated Wastes D006	Digestion/AA	EPA Method, (Appendix C-8)
Chromium Contaminated Wastes D007 F006, F007 K062, F016	Digestion/A	EPA Method 218.1 (Total Chromium) EPA Method 7196 (Hexa-valent Chromium)
Lead Contaminated Wastes D008, K049-K052	Digestion/AA	EPA Method 239.1
Mercury Contaminated Wastes D009	Digestion/AA	EPA Method (Appendix C-8)
Selenium Contaminated Wastes D010	Digestion/AA	EPA Method (Appendix C-8)
Lindane Solution D013	Gas Chromatography	EPA Method (Appendix C-8)
Toxic Organics U044, U122, U138, U220, U080, U188, U201, U210, U239, U225, U228, U021, P105, F003, F005, U002, U019, U112	Gas Chromatography	EPA Method (Appendix C-8)

TABLE C-6

RATIONALE FOR WASTE CHARACTERIZATION TESTING

Waste	Test Parameter	Rationale
D001	Flashpoints	Flash points of ignitable wastes are less than 140°F
	EP Toxic (Pb,Cr)	Paint wastes may contain Pb or Cr pigment
D002	pH	Determines reactivity and treatment needed
	Buffer Capacity	Determines treatment
	Cloride	Determines treatment method.
	Cyanide	Determines reactivity[1]
	Metals	Determines treatment
F001, F002	Cyanide	Determines reactivity[1]
	Flashpoint	Determines ignitability
	pH	Determines reactivity
F003, F005	Flashpoint	Determines ignitability
F006	pH	Determines reactivity
	Cd,Cr,Ni	Determines treatability
D013	Sp. Gravity	Determines concentration of lindane in wastewater
K052	Metals	Determines disposal options
	Cyanide, Sulfide	Determines reactivity[1]
K062	Hexavalent Cr,Pb	Either of these two may be present
P105	pH	Determines reactivity[1]
	Flashpoint	Determines ignitability
	Cyanide, Sulfide	Determines reactivity[1]

---

1 If a waste is determined to be reactive, it is not accepted at PROTECO for treatment, storage or disposal.

TABLE C-7

PARAMETERS AND TEST METHODS

Parameter	Test Method	Reference
Arsenic	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Barium	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Cadmium	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Chromium	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Fluoride	Colorimetric or Potentiometric	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Lead	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Mercury	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Nitrate (as N)	Colorimetric Method 352.1	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Selenium	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Silver	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Lindane	Gas Chromatograph	Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in water and Wastes
Chloride	Colorimetric	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
Iron	Atomic Absorption or Phenanthroline Method	Standard Methods for the Examination of Water and Wastewater, 14th Ed., American Public Health Association, 1975
Manganese	Atomic Absorption	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.

TABLE C-7 (CONTINUED)

PARAMETERS AND TEST METHODS

Parameter	Test Method	Reference
Phenols	Gas Chromatograph Method 8.04	Test Methods for Evaluating Solid Waste. Physical/Chemical Methods, USEPA SW-846.
Sodium	Flame Photometric Method	Standard Methods for the Examination of Water and Wastewater, 14th Ed., American Public Health Association, 1975
Sulfates	Colorimetric	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983.
pH	Electrometric	Test Methods for Evaluating Solid Waste. Physical/Chemical Methods, USEPA SW-846.
Specific conductivity	Method 120.1	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-790020, March 1983.
Total organic carbon	Combustion, Methods 415.1	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-790020, March 1983.
Total organic halogens	Method 450.1	USEPA, Office of Research and Development, Environmental Monitoring and Support Laboratory, EPA-600/4-81-056, November 1980
Coliforn Bacteria	Standard Plate Count	Standard Methods for the Examination of Water and Wastewater, 14th Edition, American Public Health Association, 1975.
Paint Filter	Method 9095	Test Methods for Evaluating Solid Waste. Physical/Chemical Methods, USEPA SW-846.

using stabilization/fixation treatment method. The test will be useful in determining changes in waste characteristics by contamination of the waste with halogenated solvents and the accuracy of the WPS. Compatibility of the waste to the landfill liner system can be determined from this data. This test can also be used to determine whether the material is acceptable for off-site incineration.

- Chloride -- This will be used to determine the presence of chlorides in the waste. The parameter will be characterized in aqueous wastes, slurries and sludges. These tests will be useful in determining if the waste can be treated by the stabilization/fixation treatment process, since chloride, though much less toxic than many other materials, is difficult to bind by this method.

- Percent Acidity -- determines the acidity in the waste by species. It is only used if the waste is aqueous and below a pH of 7.

- Percent Alkalinity -- determines the amount of alkalinity in the waste by species. It is only used if the waste is aqueous and above a pH of 7.

- Stabilization/Fixation -- is run to determine whether the waste is amenable to stabilization/fixation and to determine the ratio for each waste required to effect stabilization/fixation.

- Liquid Waste Compatibility -- is tested to determine whether liquid wastes stored or processed together are compatible.

- Paint Filter Test -- is used to indicate if free liquid is present in a solid or semi-solid material.

C-3b <u>Test Methods and Parameter Tolerance Limits</u>

Table C-7 lists the test methods that will be used by PROTECO to measure each of the analytical parameters described in Section C-3a. The

table also lists a reference source for each test method. A copy of each of the test methods used by the site that are not a standard EPA test method as found in either "Test Methods for Evaluating Solid Waste" or "Methods for Chemical Analysis of Water and Wastes" are provided in Appendix C.2.

It is not possible at this time to detail the QA/QC procedures which will be employed by PROTECO's new laboratory facility since not all of the manufacturer's equipment operation manuals have been obtained. Upon final receipt of the equipment and manuals, a QA/QC plan will be developed following the sample plan given in Appendix C.6. This plan will be available on-site for inspection during the lifetime of the facility and will be updated to include each new piece of equipment obtained. The inspection schedule for the laboratory facility is given in Table F-1 and the training involved for laboratory personnel is given in Section H.

The only parameter which has a tolerance limit established for the stabilization/fixation operation at PROTECO are those wastes containing organic acids. Generally, waste mixtures which contain over one percent of organic acids will not be stabilized.

The process tolerance limits for acceptability of chlorides and halogens cannot be specified at this time. Instead, each waste or waste mixture will be tested individually on a case-by-case basis. The tests which will be conducted will be to establish that the wastes are chemically fixed and will follow the EPA Toxic Characteristics Leachate Procedure, which is scheduled for promulgation on November 8, 1986.

The process tolerance limits which will be used will be established after the EPA tolerances for chlorides and halides are announced. This announcement is scheduled for November 8, 1986.

C-3c <u>Sampling Methods</u>

Proper sampling is necessary to obtain reliable information for the management of hazardous wastes. Hazardous wastes arrive at the site in

variety of containers, devices and quantities. The sampling methods used by PROTECO will ensure that a representative sample of waste is obtained.

C-3c(1) Obtaining Representative Samples from Generators. Each generator interested in having hazardous wastes treated, stored or disposed of at the PROTECO facility will be required to provide PROTECO with a representative sample of their waste. The generator will also be required to submit a PROTECO Generator Sample Form as provided by PROTECO along with the representative sample. The generator will be required to sign the certification at the bottom of the form. A copy of the Generator Sample Form that will be used and the instruction sheet are provided in Figure C-5. Additionally, the PROTECO chemist will review the sampling method to ensure that it is correct.

C-3c(2) Procedures for Collecting Representative Samples at PROTECO. Hazardous wastes will be delivered by outside transporters or by the PROTECO transportation staff to the facility for treatment, storage and/or disposal by any one of the following methods:

- Bulk Deliveries
  - Liquids
    - 10,000 gal. tanker truck
    - 8,000 gal. tanker truck
    - 3,000 gal. vacuum truck
  - Solids and Sludges
    - 8, 16, 22, 30 or 40 yd^3 dump trucks
- Containerized
  - Liquids
    - DOT approved containers that may vary in volume capacity from several milliliters to possibly 100-gallons. Most deliveries, however, will be contained by either 55-gallon metal or polyethylene drums.

FIGURE C-5

GENERATOR SAMPLE FORM

Sample No. _____

Return this sheet completed and signed with a 1000-ml representative sample to the following address:

PROTECCION TECNICA ECOLOGICA INCORPORATED
Firm Delivery
Ponce, Puerto Rico 00731.

Name of Generator _____

Address of Generator _____
street P.O. Box

_____
city state zip code

Name of Contact _____

Telephone Number (____)_____

Generator's EPA I.D. Number _____

Collector _____ Date _____ Time _____ Hours

Purpose of Sampling _____

Type of Sample _____

Type of Process Producing Sample _____

_____

Field Information (including sampling equipment and sampling methodology used) _____

_____

_____

_____

_____

_____

I believe that the information submitted herein is true, accurate and complete.

Signature _____ Date _____
authorized personnel

PROTECO Approval of Sampling Method

Chemist _____ Date _____

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FIGURE C-5 (CONTINUED)

PROTECCION TECNICA ECOLOGICA, INCORPORATED

WASTE APPROVAL SHEET

---

1. Complete to the best of your knowledge an PROTECO Waste Characterization Sheet. Do not leave any spaces on the sheet blank. If a question is non-applicable, please put N/A in the blank.

2. Collect, package, and label for shipment and analysis one 1000-ml (approximately two pints) representative sample of each waste to be considered. Please be sure to attach a label to each sample container. The label should include the sample collector's name, the date and time the sample was collected, the generator's sample number, and the generator's name and address.

3. Include in the same package with each sample a completed and signed copy of the attached Generator Sample Form.

4. Return the original completed and signed waste Characterization Sheet along with the representative sample and the Generator Sample Form to the following address:

PROTECO
Firm Delivery
Ponce, Puerto Rico 00731.

Note: The sampling method you use must meet federal guidelines and your sample must be representative. Call us before sampling if you are unsure what to do.

- Sludges

  - DOT approved containers that may vary in volume capacity from several milliliters to possibly 100-gallons.

    Most deliveries, however, will be contained by either 55-gallon metal or polyethylene drums.

- Solids

  - Approved DOT cardboard and/or metal containers of varying capacities with most such deliveries contained in 55-gallon containers.

Only wastes in DOT approved transportation containers are accepted at the site. Representative samples will be collected by PROTECO upon receipt of the waste at the site. Certain procedures and equipment have been classified by EPA as suitable for obtaining representative samples of hazardous waste. Table C-8 lists the sampling equipment that will be used by PROTECO to sample various types of waste, based on the information provided by EPA. Appendix C.3 shows the sampling equipment used and describes proper sampling procedure.

Random sampling methodology will be used in collecting the representative samples from the containerized waste received in each waste shipment. Ten percent (10%) of the containers holding each specific waste type that are received in each shipment will be analyzed by PROTECO. All containers shall be grounded prior to the beginning of the sampling process if the receiving container is made out of metal. The random sampling procedures and the random number table that will be used by PROTECO are provided in Appendix C.4.

All hazardous wastes shipped by either vacuum truck or tanker will have representative samples collected when they reach the facility. Grab samples will be pulled through each accessible hatch using a weighted bottle sampler. A composite sample made from the combined grab samples

TABLE C-8

SAMPLING EQUIPMENT FOR VARIOUS TYPES OF WASTE

Waste Type	Sampling Equipment	Additional Information
Ignitable or chlorinated wastes in drums, barrels and similar containers	COLIWASA (glass)	Not for containers greater than 5 ft. deep.
Corrosive wastes in drums, barrels, vacuum trucks and similar containers	COLIWASA (plastic)	Not for containers greater than 5 ft. deep.
Wastes transported by tanker trucks	Weighted bottle sampler	May be difficult to use on very viscous liquids.
Sludges in drums, barrels, dump trucks or similar containers	Trier	Waste should be moist or sticky.
Powdered or granular solids in bags, drums, barrels or similar containers	Thief	Sample particles should be 0.6 cm in diameter or less.
Large grained solids in bags, drums, barrels or similar containers	Large trier	May incur difficulty in retaining core of very dry granular materials.
Wastes received in containers of one (1) gallon or less.	Pipette	Not for containers larger than one gallon. May be difficult to use on viscous materials.

Source of Information
"Samplers and Sampling Procedures for Hazardous Waste Streams," EPA600/2-80-018.

will be analyzed. These grab samples will be taken from a range of depths to compensate for any separation of phases.

Hazardous wastes received in dump trucks would be sampled as a sludge or a solid since free liquids are not transported by this method. Depending on the waste consistency, a sample would be collected using either a sample thief or trier. One sample would be withdrawn from at least three different points including the back, middle and front of the dump truck. Also, samples will be taken from a variety of depths if phasic wastes are present (the presence of phasic wastes is determined by slow insertion of a glass sampling rod, sealing of the top and bringing the rod into visibility). These samples will be combined to form a representative composite sample of the waste.

Most of the hazardous wastes that are received at the facility in containers of one gallon or less are known "commercial chemical products" as described under 40 CFR 261.33(f). They are received at the facility due to off-specification conditions or because the generator has discarded the "material". Such wastes are sampled using a pipette.

Table C-9 lists the recommended sampling points for the types of wastes received at the facility and Table C-10 describes the methodology that will be used by PROTECO sampling personnel to insure that a representative sample is collected.

Table C-11 describes the sample containers that will be used by PROTECO sampling personnel whenever representative samples are collected from the wastes received by the facility. All precautions will be taken to ensure that the sample containers are inert to the waste being collected. Since glass is inert to most hazardous waste, it will be used most often.

Before sampling any incoming hazardous waste, the PROTECO sampling personnel will typically wear the following personnel protective equipment:

TABLE C-9

SAMPLING POINTS FOR WASTE CONTAINERS

Container Type	Sampling Point
Drum, bung on one end	Withdraw sample through the bung opening.
Drum, bung on side	Lay drum on side with bung up. Withdraw sample through the bung opening.
Barrel, fiberdrum, buckets, sacks, bags	Withdraw samples through the top of barrels, fiberdrums, buckets and similar containers. Withdraw samples through fill openings of bags and sacks. Withdraw samples through the center of the containers and two different points diagonally opposite the point of entry.
Vacuum truck and similar containers	Withdraw sample through open hatch. Sample all other hatches.
Dump truck	Withdraw samples through at least three different points near the top of pile to 2 points diagonally opposite each other.
Tank trucks	Sample from the top through the sampling hatch. With- draw sample from top, middle and bottom of tank.

* Source of Information

"Samplers and Sampling Procedures for Hazardous Waste Streams", EPA-600/2-80-018.

TABLE C-10

METHODS USED TO SAMPLE HAZARDOUS WASTES

Sampling Method	Description of Sampling
Sampling a Drum	
1. Position drum so bung is up. 2. Allow contents of drum to settle. 3. Slowly loosen the bung with a bung wrench. 4. Allow gas pressure to be released. 5. Remove bung and collect sample with Coliwasa through bung hole.	Prepare a representative composite sample using the Coliwasa sampler by taking three (3) grab samples from bottom, middle and top of the drum.
Sampling a Vacuum Truck	
1. Open the tank hatch. 2. Collect a sample with a Coliwasa. 3. When necessary, take a sediment sample.	Prepare a set of samples collected from the liquid sample and the sediment sample taken from the vacuum truck.
Sampling a Tanker or Storage Tank	
1. Open the tank hatch. 2. Collect samples from the tanker with a weighted bottle sampler.	Prepare three separate samples using the weighted bottle sampler by taking three (3) grab samples from bottom, middle and top of the tanker.
Sampling a Barrel, Fiberdrum, or Bags	
1. Position the container in an upright position. 2. Open the container slowly. 3. Collect a composite sample using either a sampling thief or trier depending on waste consistency.	Prepare a representative composite sample from three (3) grab samples from bottom, middle and top of container using either the thief or trier.
Sampling a Dump Truck	
1. Determine waste consistency. 2. Determine the sampling points and angles (front, middle and back of truck). 3. Collect samples using sampling trier	Prepare a representative composite sample by withdrawing samples through the three different points near the top of the pile and two points diagonally opposite each other.

*Source of Information:

"Samplers and Sampling Procedures for Hazardous Waste Streams", EPA600/2-80-018

(0059c)

TABLE C-11

SAMPLE CONTAINERS USED FOR VARIOUS TYPES OF WASTE

Waste Type	Example of waste	Sample Container
Oil waste	Hydraulic oil	1 Qt. glass
Halogenated organics	Methylene chloride	1 Qt. glass or amber glass
Non-halogenated organics	Toluene	1 Qt. glass
Phenolic wastes	Phenol	1 Qt. glass
Photosensitive wastes	Tetrahydrofuran	1 Qt. amber glass
Metal bearing wastes	Electroplating sludges	1 Qt. polyethylene
Acidic wastes	Sulfuric acid	1 Qt. polyethelene
Alkaline wastes	Sodium hydroxide	1 Qt. polyethylene
Pesticide wastes	Lindane solution	1 Qt. glass
Ignitable wastes	Acetone	1 Qt. glass

- disposable, chemical-resistant coveralls
- chemical resistant gloves and boots
- safety glasses or goggles
- chemical-cartridge respirator

After the grab samples have been taken and the composite sample prepared, the composite sample will be labeled and then logged into the PROTECO laboratory analysis log book.

C-3d Frequency of Analysis

As each generator's representative sample is received by PROTECO it will be analyzed for a number of parameters as described in Table C-4. Parameters that are analyzed will be selected by the facility chemist using Table C-4, the hazard classification of the waste and the information provided by the generator of the WPS as references. The result of these original analyses are noted in the Standard Reference Form (Figure C-4). In general, all parameters in Table C-4 will be run for incoming load fingerprint testing, unless the waste type indicates that the testing is not applicable (example: testing viscosity or pH of a solid).

All wastes received by PROTECO in a vacuum truck, tanker, or dump truck will be subject to incoming load fingerprint testing upon receipt. Ten percent (10%) of the containers of each specified waste type received in each waste shipment will be routinely fingerprint tested by PROTECO. Random sampling methodology will be used to choose the containers to be sampled. Any time PROTECO analyzes a waste that is received in a waste shipment, the laboratory will analyze those parameters chosen by the facility chemist based on historical and analytical knowledge of the waste. The results of this analysis will be recorded on the Incoming Load Sample Result Form (Figure C-6).

The results of the analysis of the waste shipment will be compared to the results of the original analysis performed on the generators representative sample as listed on the Standard Reference Form (Figure C-4). Table C-12 lists the frequency of analysis as described above.

FIGURE C-6

INCOMING LOAD
SAMPLE RESULT SHEET

Sample No. _____

Generator Name: _____
Address: _____
City: _____ State: _____ Zip Code: _____

Sampler's Name: _____
Location of Sampling: _____
Date: _____ Time: _____

Waste to be Analyzed: _____
EPA Code No.: _____ Manifest No.: _____
DOT Labels: _____

Field Information (Sampling methodology, weather): _____
_____
_____

Physical Characteristics

Physical State: _____ Color: _____
Odor: _____ Texture: _____
Specific Gravity: _____ Viscosity: _____

Chemical Characteristics
(See Standard Reference Form)

pH: _____ Flash Point: _____ Water Content: _____
Cyanide _____ Halides: _____
Oxidizable Organics: _____

Waste Accepted or Rejected: _____

Signature of Laboratory: _____

(0059c)

TABLE C-12

FREQUENCY OF ANALYSIS

Type of Shipment	Frequency
Vacuum Truck	Every shipment received by the facility.
Tanker Truck	Every shipment received by the facility.
Dump Truck	Every shipment received by the facility.
Flatbed with drums, barrels or similar containers	10% of the containers for each specific waste received in each shipment.
Semitruck with drums, barrels or similar containers	10% of the containers for each specific waste received in each shipment.
Representative Sample sent by a generator	All samples received by the facility.

C-3e Procedures for Recharacterizing Wastes

As required in 40 CFR 264.13 (a)(3) an analysis must be repeated as necessary to ensure that it is accurate and up to date. A generator is to inform the facility of any changes in his/her waste due to process or operational changes or any changes in raw materials. In addition to this agreement, PROTECO will send a notification letter annually to any generator whose hazardous wastes are accepted for treatment, storage or disposal. This form will be completed, signed by the generator and returned to PROTECO. The form will be kept at the site laboratory for the operating life of the facility and will be filed in the generator's folder. A copy of the notification letter is provided in Figure C-7.

If changes are noted in the letter, PROTECO will request a new WPS and representative sample from the generator. The new representative sample will be analyzed by the PROTECO laboratory following the pre-acceptance testing methodology. The procedures described in Section C-2b(1), Initial Waste Characterization, will be utilized to determine if the "new" waste will be accepted.

C-3f Additional Requirements for Ignitable, Reactive or Incompatible Wastes

If ignitable wastes are treated, rather than sent off-site for resource recovery, these wastes will be tested to assure adequate treatment by the stabilization/fixation process, as described in Section C-3a (3)b, Process-Specific Waste Testing Parameters. The product and leachate will be tested to confirm non-ignitability.

C-4 Screening and Evaluation of Waste Shipments [40 CFR 270.14(b)(3) and 264.13(c)]

C-4a Procedures for Characterizing Waste Shipments

PROTECO's objectives in characterizing each waste shipment received at the facility will be to document consistency between the waste and the manifest and to assure that the waste received from a generator is the

FIGURE C-7

GENERATOR NOTIFICATION LETTER

(DATE)

Generator Name
Generator Address

Dear (Contact Name)

Attached you will find a Generator Notification Form. PROTECO requests that all generators whose hazardous waste is treated, stored or disposed of at the PROTECO facility complete and return the attached form to the Penuelas office within 30 days of the receipt of this letter. The form should be filed out by persons knowledgeable of plant processes, operational procedures and the wastes generated from each process.

In order for PROTECO to continue handling the treatment, storage or disposal of your hazardous wastes it is necessary for you to return a completed form to the office. If the form is not received or you have not contacted the PROTECO office within 30 days of receipt of this letter, PROTECO will no longer be able to treat, store or dispose of your hazardous wastes.

If you have any questions, please feel free to call the Lab Manager at (809) 836-2058. We are looking forwarded to hearing from you.

Very truly yours,

Jorge Fernandez
President

FIGURE C-7 (CONTINUED)

PROTECCION TECNICA ECOLOGICA, INC.
FIRM DELIVERY
PONCE, PUERTO RICO 00731

Generator Notification Form

Generator EPA ID No.: _____

Generator Name: _____

Generator Address: _____

_____

_____

Generator Contact: _____

****

1. Have any changes occured during the last year or do you expect any changes to occur in the future that have affected or could affect the plant processes?

   Yes _____(Go to 2)
   No _____(Go to 3)

2. Please describe these changes and how they could or have affected the wastes generated by your facility.

   _____
   _____
   _____
   _____

3. Have "new" raw materials or chemical products been substituted or added to an existing plant processes?

   Yes _____(Go to 4)
   No _____(To to 5)

4. Please list all the new materials that have been substituted or added to an existing plant process.

   _____
   _____
   _____

5. Has or does the facility plan to add or develop a new plant process to manufacture products?

   Yes _____(Go to 6)
   No _____(Go to 7)

6. Please describe the new plant process and the waste that may be or are being generated from the operation of this plant process.

   _____
   _____
   _____

FIGURE C-7 (CONTINUED)

PROTECCION TECNICA ECOLOGICA
FIRM DELIVERY
PONCE, PUERTO RICO 00731

Generator Notification Form (Continued)

7. Have operational procedures been altered that could result or have resulted in an increase or decrease in the chemical components (i.e. composition) in the waste itself?

Yes _____(Go to 8)
No _____(Go to 9)

8. Please describe the changes that affected the chemical composition of the waste and describe the new chemical composition of the waste, listing chemical components and their percentages

_____
_____
_____
_____
_____
_____

9. Have any new operational procedures been established or are expected to be established that could result in the generation of a new waste stream or a change in the chemical composition of the old waste stream?

Yes _____(Go to 10)
No _____(Go to 11)

10. Please describe the new operational procedure and the wastes that might be generated from this operation including information on chemical components and their percentages.

_____
_____
_____
_____
_____

11. Please describe any other changes that have occurred in the last year that have affected the types or quantity or waste being generated at the facility including changes in waste component ranges.

_____
_____
_____
_____
_____

12. Does your facility expect an increase or decrease in the waste generated in the coming year?

Increase _____
Decrease _____

I believe that the information submitted herein is true, accurate and complete.

_____ _____
(Signature) (Date

(0060c-5)

waste the facility expected to receive. Although PROTECO requires all generators to notify PROTECO of changes in a waste, PROTECO will implement a variety of screening tests, as described in the waste analysis plan in Section C-3, on a routine basis to assure fulfillment of these objectives.

C-4b Waste Shipment Screening Procedures

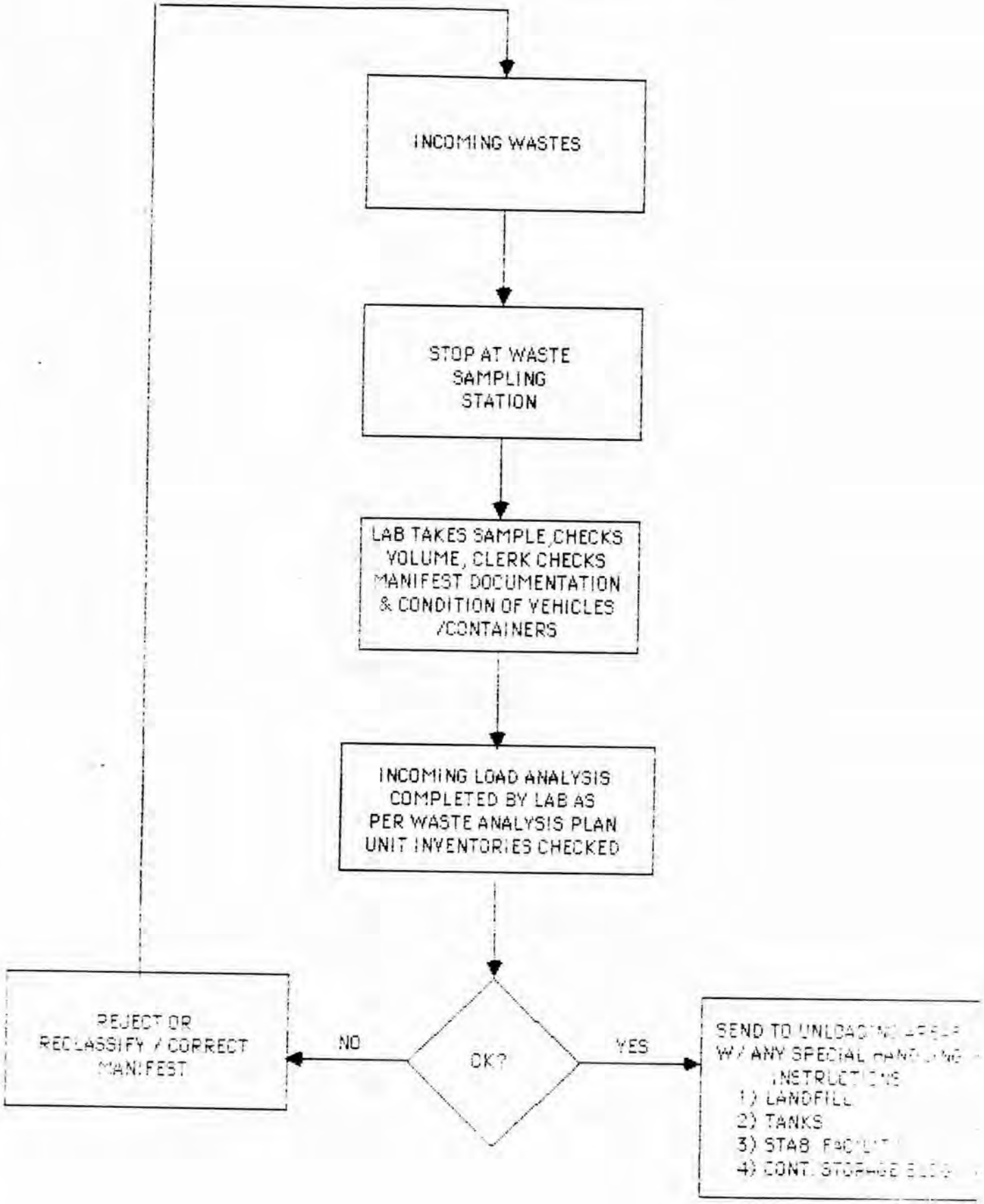
Each shipment received at PROTECO is subject to a standard procedure for waste acceptance. These procedures are outlined in Figure C-8.

PROTECO's first objective is to note any manifest discrepancies. A manifest discrepancy is a difference between the quantity, including differences in weight of a shipment or in a number count of containers, or the type of hazardous waste designated on the manifest and the quantity or type of hazardous waste a generator receives. The detection of manifest discrepancies will be noted by visual inspection, weighing or unit count and chemical analyses performed by the PROTECO laboratory. PROTECO will count all discrete units (i.e., containers) in the shipment or record the volume of entire shipment depending on the type of containment used and the waste description on the manifest. The number of containers or the volume of the shipment will then be checked against the manifest.

As described in the preamble to the Federal Regulations on hazardous waste, the detection of manifest discrepancies in type is intended to "have facilities flag obvious differences in waste type... as opposed to more subtle changes, such as parts-per-million variations in the concentrations of heavy metals within a sludge". Differences in type are, therefore, noted by PROTECO using visual inspection and rapid incoming load fingerprint analyses as previously described that are performed during and after the collection of a representative sample. Ten percent of containers for each specific waste type received in a shipment, accessible ports on a tank truck or vacuum truck, and all dump trucks containing bulk sludges or solids are visual inspected and analyzed for the following items at a minimum:

FIGURE C-8

INCOMING WASTE PROCEDURES



- Physical state of the waste
- Color
- Flashpoint
- Odor
- Texture
- Specific Gravity or Viscosity
- Free Liquids/Water Content
- Cyanides
- Sulfides

The representative composite samples are collected and labeled as described in Section C-3c and a laboratory number is given to the sample. A copy of the results obtained from the analyses performed will be kept in the laboratory on a Sample Result Form (Figure C-6).

C-4c Procedures for Screening Restricted Wastes

PROTECO does not accept reactive wastes for treatment, storage or disposal at the facility. Therefore, PROTECO will analyze those wastes that have potential for reactivity due to fact that they may be cyanide or sulfur bearing wastes which, when exposed to pH conditions between 2 and 12.5 can generate toxic fumes, gases or vapors. In general, all waste shipments will be screened for cyanide and sulfide unless the test is clearly not applicable.

C-4d Procedures for Accepting or Rejecting Waste Shipments

PROTECO rejects waste shipments for the following reasons:

- the weight of the waste shipment or the number of containers in a waste shipment is found to be inconsistent with the manifest information

- the waste shipment is a restricted waste or includes waste units that contain a restricted waste (i.e., a reactive waste) due to unacceptable levels of cyanide or sulfide

- the analyses performed indicate significant off-specification conditions (i.e., change in viscosity, specific gravity, pH, free liquids)

The waste shipment will be initially recorded by volume or the containers counted. PROTECO will allow a discrepancy limit of ten percent in weight of the manifested volume for bulk shipments. If there is a discrepancy of one container in the waste shipment, PROTECO will contact the generator and try to resolve this discrepancy. If the discrepancy in volume or number of containers cannot be resolved between PROTECO and the generator, the shipment will be rejected and returned to the generator.

Next, PROTECO will visually inspect the waste for physical state, color, odor and texture. If discrepancies in the waste seem apparent based on visual inspection, PROTECO will postpone a discussion to reject the shipment until after the waste is screened for specific gravity, viscosity and other fingerprint parameters. If the tested parameters are within specified tolerances for the waste, the waste will be accepted; otherwise the shipment will be rejected by the facility and the generator will be contacted to resolve the problem.

Once a generator is contacted for resolution of such a problem, PROTECO will advise the generator that the waste has been rejected for one of the following reasons:

- a discrepancy in the volume

- a discrepancy in the number of containers as listed on the manifest

- analysis of the representative samples collected by PROTECO is not consistent with historical information.

PROTECO will resample and reanalyze the waste only if discussions with the generator present strong evidence that the waste has not changed and

that errors in shipment are unlikely. If further testing does not resolve the discrepancy, the waste must be rejected and recharacterized as a new waste. The decision to reject a waste is made by the Lab Manager.

C-5 Waste Analysis Plan for Containers (40 CFR 264.172, 264.175 and 264.177)

C-5a Procedures for Assuring Compatibility of the Waste with Container Materials

To prevent premature deterioration of a container it is necessary for the wastes stored at the facility to be compatible with the container and the container liner material. All hazardous wastes accepted by PROTECO for treatment, storage or disposal at the facility are originated by a generator at his/her location. At the time of initial waste stream approval, PROTECO will recommend to the generator the container and/or liner that would be compatible with the waste based on the information provided in Table C-13. PROTECO will also record the container type on the Standard Reference Form.

PROTECO visually inspects all containers in a waste shipment when they are received and accepted at the facility checking for obvious signs of deterioration or leakage. In addition, PROTECO accepts only those containers for transport to the facility that are DOT-approved containers. As described in Section F, PROTECO also visually inspects all containers stored in the drum storage area on a weekly basis, again checking for signs of container deterioration, leakage or pressurization.

C-5b Container Storage Facility Waste Movement Procedures

Standard procedures have been developed for receiving wastes at the container storage facility (Figure C-9). After incoming waste acceptance procedures at the PROTECO gate, the truck is directed to the container storage building.

TABLE C-13

COMPATIBILITY CHART: CHEMICALS VERSUS CONSTRUCTION AND LINING MATERIALS

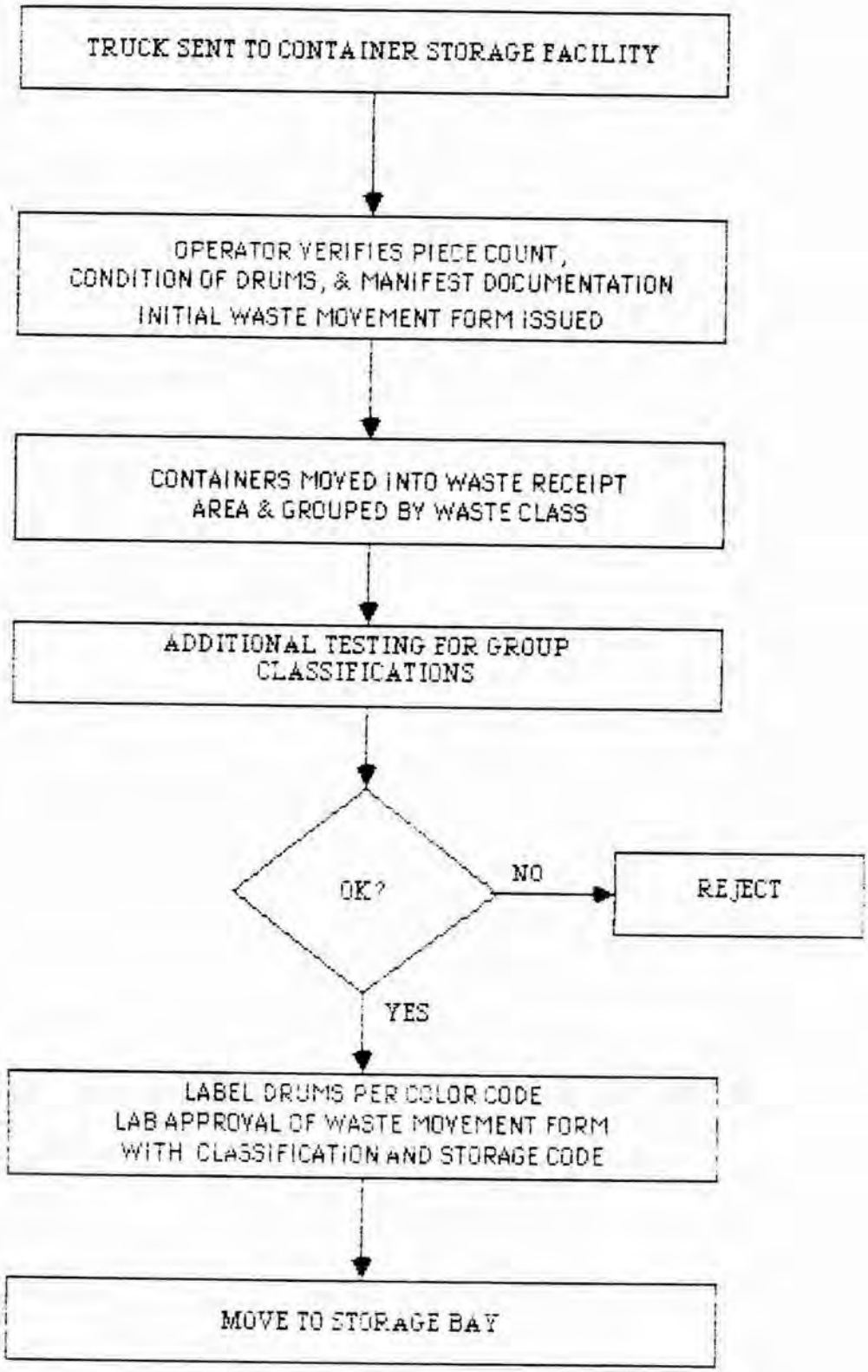
Construction Material	Chemicals Incomptible With
Steel	Mineral and Organic Acids: nitric, hydrochloric, sulfuric acids
Aluminum	Alkalies: potassium hydroxide, sodium hydroxide, mineral and organic acids
Magnesium	Mineral and organic acids
Lead	Acetic acid, nitric acid, hydrochloric acid, alkalies
Copper	Nitric acid, ammonia, hydrochloric acid, sulfuric acid
Nickel	Nitric acid, ammonia, hydrochloric acid
Zinc	Hydrochloric acid, nitric acid, sulfuric acid, alkalies
Tin	Organic and mineral acids, alkalies
Titanium	Sulfuric acid, hydrochloric acid

Lining Materials	Chemicals Incompatible With
Alkyds	Strong mineral acids, strong alkalies, alcohols, ketones, esters, aromatic hydrocarbons
Vinyls (polyvinyl-chloride-PVC)	Ketones, esters, aromatic hydrocarbons
Chlorinated Rubbers	Organic solvents
Epoxy: (amine-cured, polyamide cured, or esters)	Oxidizing acids (nitric acid), ketones
Coal Tar Epoxy	Strong organic solvents
Latex	Oxidizing acids, ketones, esters
Polyesters	Oxidizing acids, strong alkalies, mineral acids, ketones, aromatic hydrocarbons
Silicones	Strong mineral acids, strong alkalies, alcohols, ketones, aromatic hydrocarbons

(0059c)

FIGURE C-9

CONTAINER STORAGE FACILITY



The operator of the facility reinspects the waste shipment, verifies the manifest and initially issues waste movement form (WMF) shown in Appendix C.5-1. This waste movement form details all the information obtained on the entire shipment. The containers are then moved into the waste receipt section of the facility and grouped according to waste class.

At this point, the trained operator will determine, after examination of all the previously acquired information on the waste-load, whether further testing of individual containers is necessary. The operator can require all of a shipment or only certain wastes or containers to undergo further testing such as a fingerprint analysis. Any containers which show manifest inaccuracies from this second level of testing will be rejected and held while the generator and PROTECO negotiate proper handling of the waste.

After this second level of testing is complete, the PROTECO laboratory issues a waste movement form and a color coded label which state the waste classification. Each container is affixed with a label and moved to the storage bay for wastes of that classifications. A copy of the waste movement form is returned to the PROTECO Laboratory for inventory reconciliation and recordkeeping.

C-5c <u>Procedures for Characterizing Collected Leaks, Spills and Run-off</u>

PROTECO is not expected to generally experience any significant leaks or spills in the container storage area. If a leak or spill should occur, the procedures delineated in Section G, the PROTECO Contingency Plan, would be followed. In general, liquid spills would be managed as per the same waste classification as the spill producing the waste.

The proposed container storage facility will have a series of separate bays with individual sumps to collect spills and leaks. Typically the contents of the sumps will be assumed to contain a hazardous waste and the contents will be treated by one of the facilities hazardous waste treatment or storage processes.

Refer to Section D for further discussion of spill collection

(0059c)

C-5d Procedures for Assuring Compatibility of Wastes with Used Containers

Empty used containers will not be reused at the facility. In limited circumstances, solids of the same waste class may be combined as part of Container Storage Facility operations. Refer to Section D-1 for further discussion of this aspect. Used containers will either be crushed and disposed of as hazardous waste or returned to the original generator for reuse in storing the same waste stream.

C-6 Waste Analysis Plan for Stabilization/Fixation Facility [40CFR 270.14(b)(2)]

Two types of wastes will be accepted for stabilization/fixation at the PROTECO facility, liquids and solids/sludges. The handling of these wastes is outlined in Figure C-10 and Section D-9.

C-7 Waste Analysis Plan for Landfills

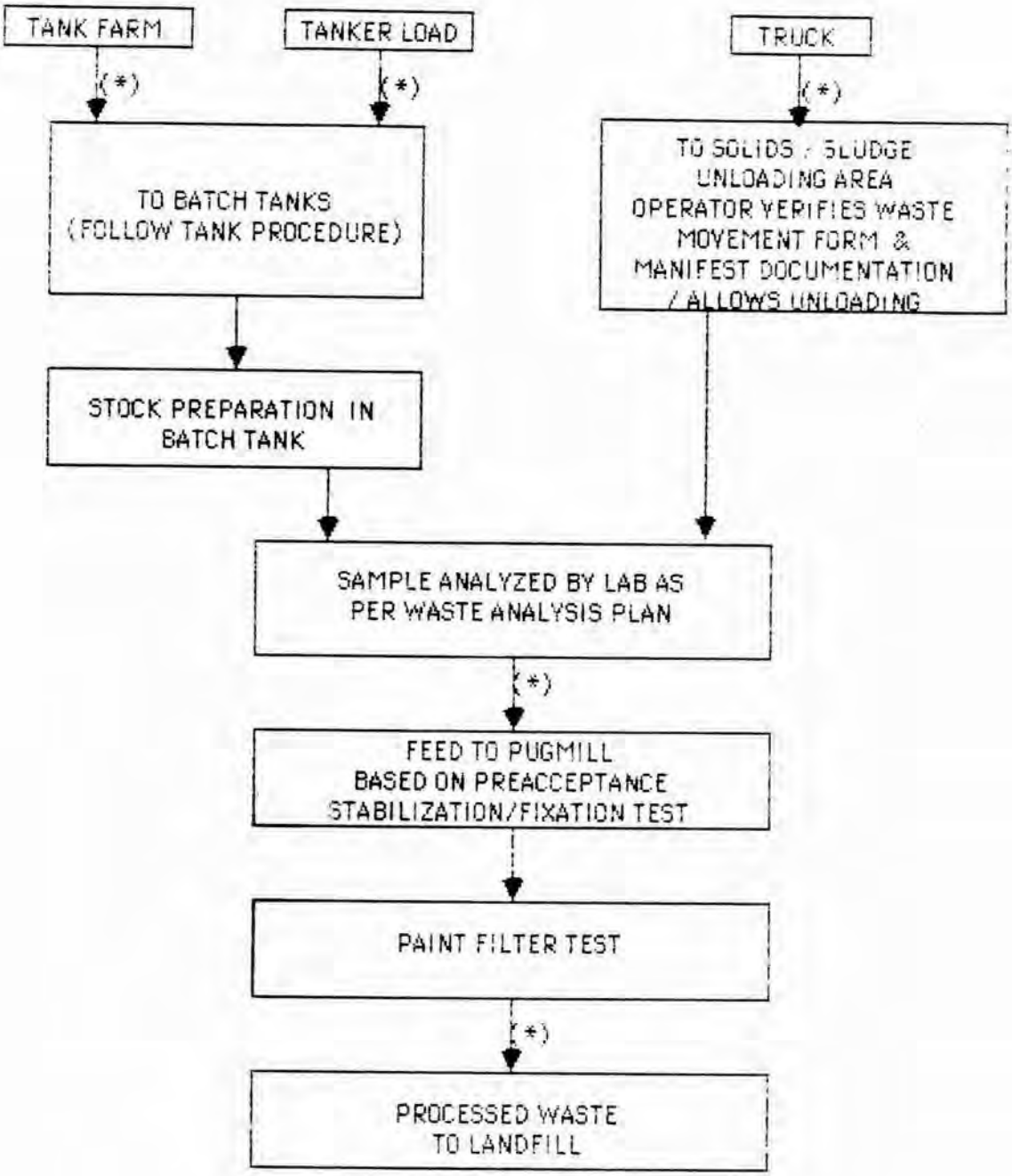
As stated previously, all wastes received at any of the PROTECO units must be accompanied by a waste movement form which has been completed and recorded at the PROTECO laboratory. This form accompanies all loads which are received at the landfill including on-site waste movements from the stabilization/fixation facility and solid wastes from the container storage facility. In this manner PROTECO ensures that no free liquids are disposed into the landfill. An outline of the waste movement procedures followed at PROTECO is shown in Figure C-11.

C-8 Waste Analysis Plan for Tanks

Waste acceptance procedures has been developed for the PROTECO tank farm to prevent mixing of any incompatible wastes or overfilling of any tank. These procedures, outlined in Figure C-12, involve a double check from the laboratory to verify the tank contents and capacity. Each waste classification has a separate color code. The key to the locking cap for the tank containing the waste has the same color code. After the operator checks the manifest and waste movement form he obtains the correct color

FIGURE C-10

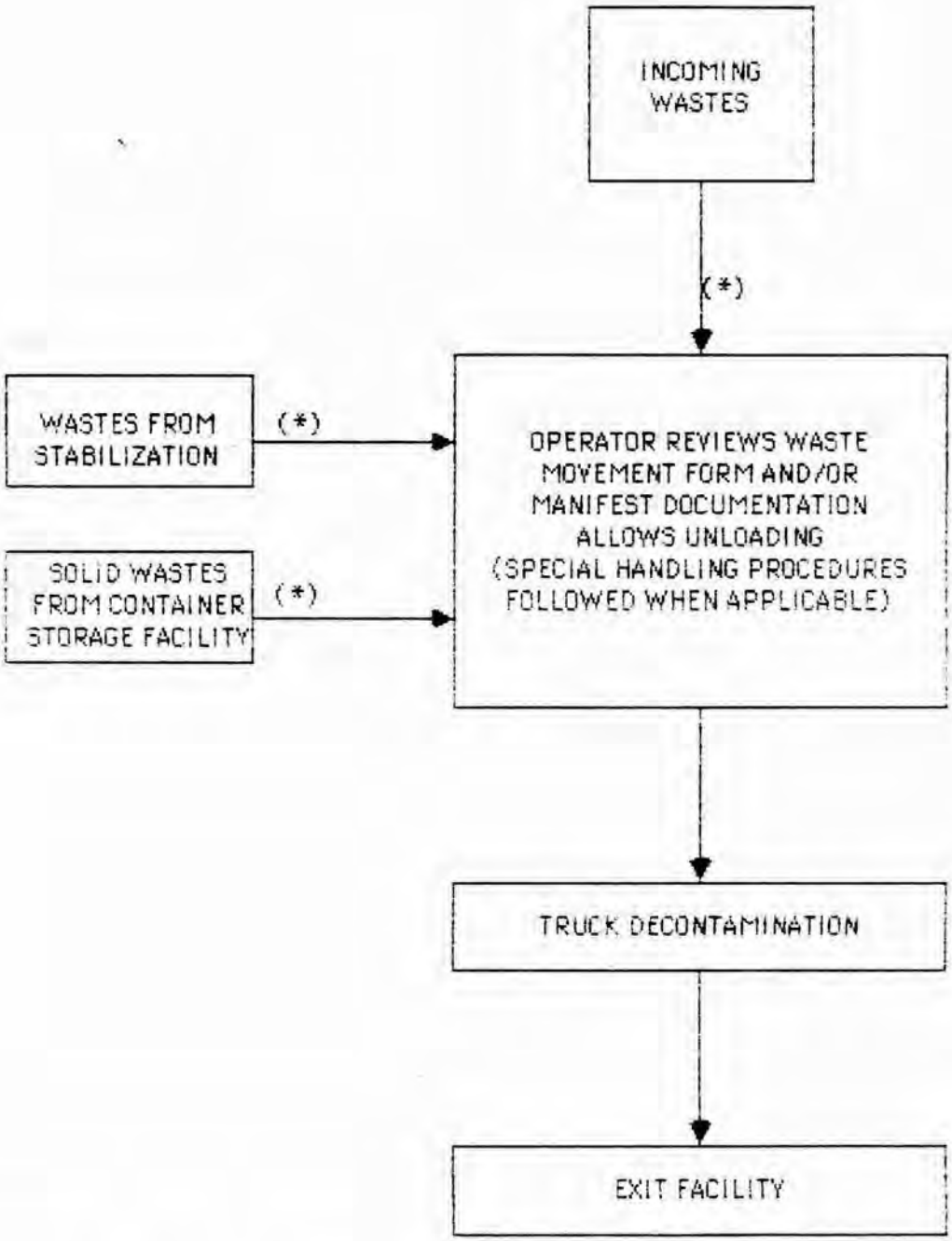
STABILIZATION/FIXATION FACILITY



(*) = WASTE MOVEMENT FORM ISSUED

FIGURE C-11

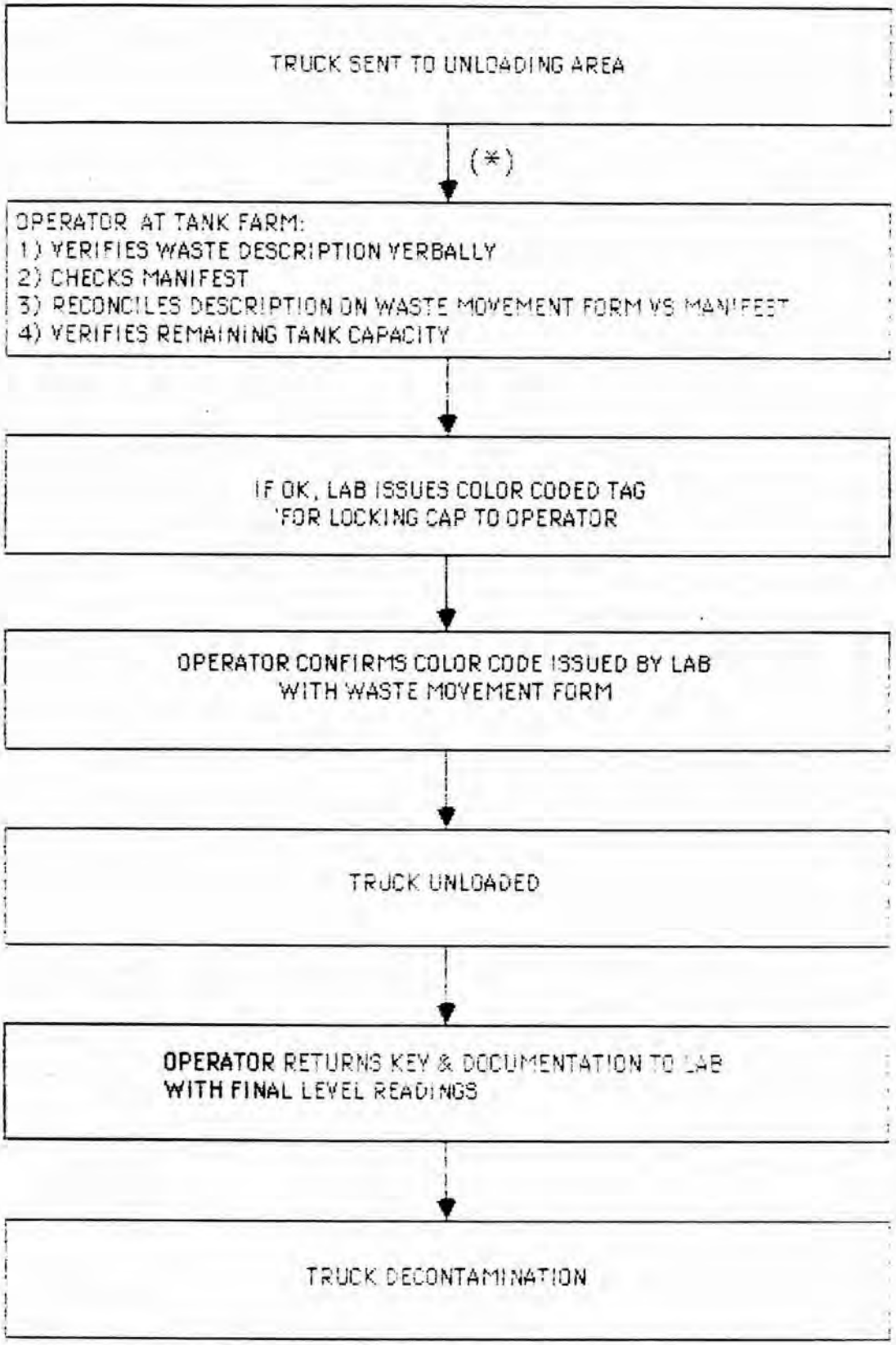
LANDFILL



(*) = WASTE MOVEMENT FORM ISSUED

FIGURE C-12

TANKS



(*) = WASTE MOVEMENT FORM ISSUED

key from the PROTECO laboratory, which verifies the remaining capacity in the tank.

C-9 Waste Analysis Plan for Drum Decant Operations

The drum decant operation has standard waste analysis procedures developed to prevent mixing of incompatible wastes and assure proper handling of all decanted solutions. This protocol is outlined in Figure C-13 and involves the obtaining of a waste movement form from the PROTECO laboratory prior to any decanting. The procedure also requires obtaining a waste movement form before any solid waste is transferred to the stabilization/fixation facility to make certain the material is non-reactive and able to be stabilized/fixed.

C-10 Waste Analysis Plan for Additional Internal Waste Transfers

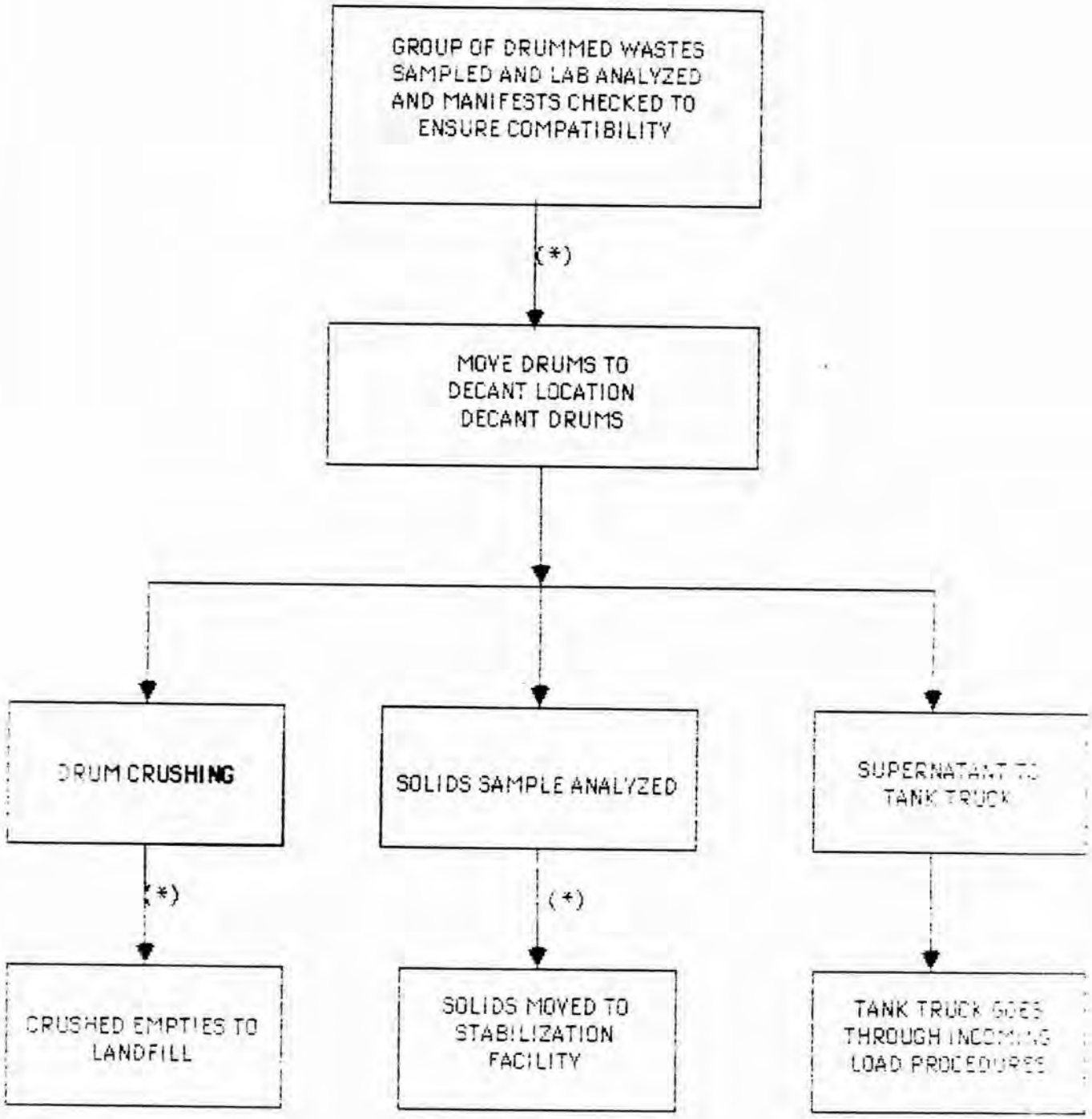
Figure C-14 outlines the waste analysis plan for other internal waste transfers which may occur at the PROTECO facility. All of these internal transfers require verification of the compatibility and proper storage or treatment of the material in the form of a waste movement form from the PROTECO laboratory. In this manner, PROTECO ensures that not only are wastes handled properly upon receipt and initial storage or treatment at the facility, the characteristics of a waste are re-evaluated prior to an internal transfer.

C-11 Testing Programs for Liners

A liner testing program is continuing at the PROTECO facility. Liner materials have been selected to meet the "Minimum Technology Standards" for liners as published by the Environmental Protection Agency. Table C-14 summarizes the status of PROTECO's liner testing program.

FIGURE C-13

DRUM DECANT OPERATIONS



(*) = WASTE MOVEMENT FORM ISSUED

FIGURE C-14

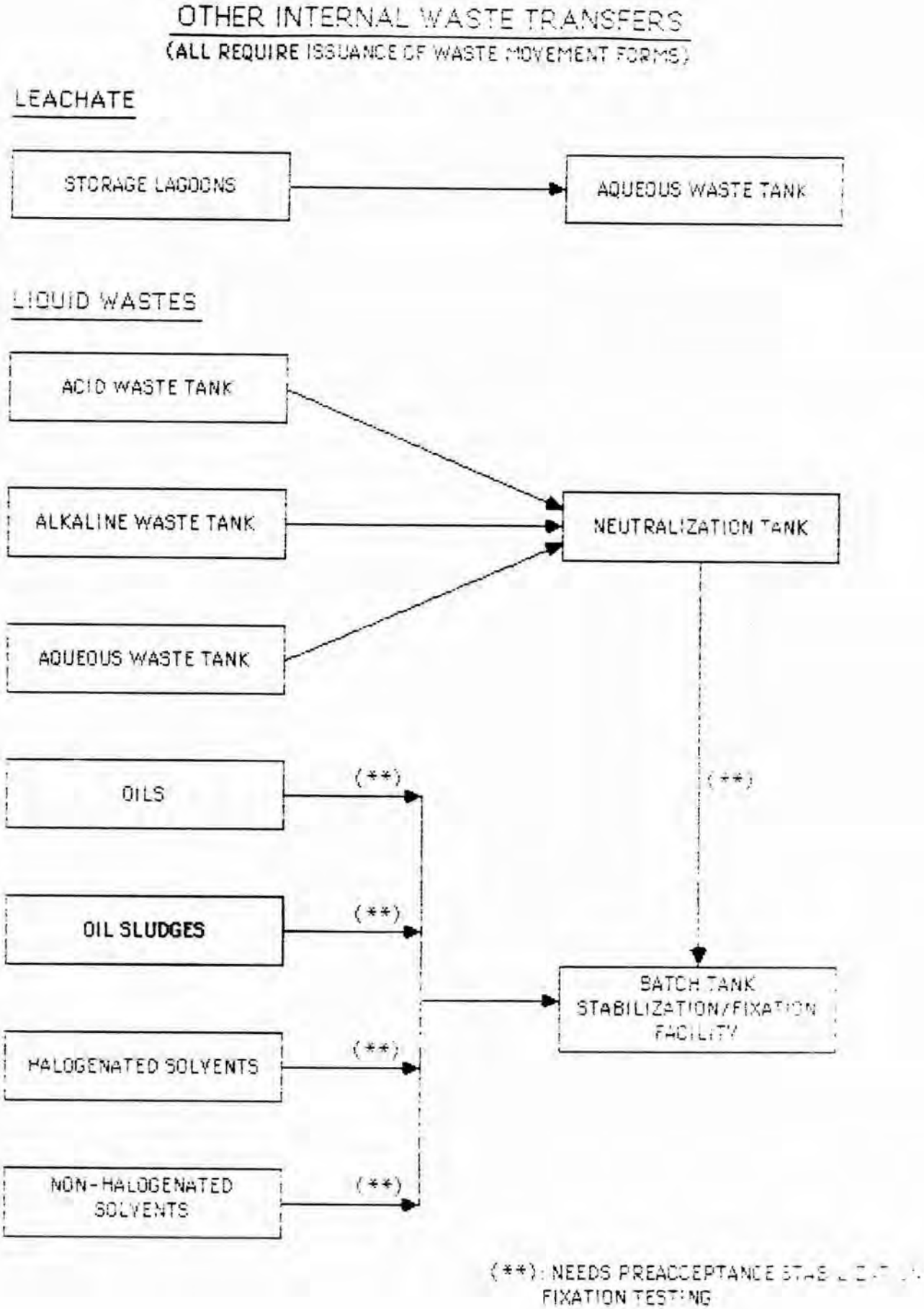

OTHER INTERNAL WASTE TRANSFERS
(ALL REQUIRE ISSUANCE OF WASTE MOVEMENT FORMS)
LEACHATE
STORAGE LAGOONS
AQUEOUS WASTE TANK
LIQUID WASTES
ACID WASTE TANK
ALKALINE WASTE TANK
AQUEOUS WASTE TANK
NEUTRALIZATION TANK
OILS
(**)
OIL SLUDGES
(**)
(**)
BATCH TANK
STABILIZATION/FIXATION
FACILITY
HALOGENATED SOLVENTS
(**)
NON-HALOGENATED
SOLVENTS
(**)
(**): NEEDS PREACCEPTANCE
FIXATION TESTING

TABLE C-14

SUMMARY OF LINER COMPATIBILITY TESTING

1) Previous synthetic liner compatibility testing using waste samples from existing wastes conducted - Data shows HDPE superior to chlorinated polyethylene (CPE)

2) HDPE has been selected as the liner material and for piping outside of lined areas, based on overall compatibility property superiority.

3) PROTECO will submit to EPA:

--vendor-supplied compatibility data for HDPE and PVC (PVC is used in the leachate collection piping)

--permeability/compatibility testing for:
  --clay materials

  --sands/gravels/crushed stones to be used in the leachate collection systems

  --filter cloths to be used in the leachate collection systems.

C-11a General Liner Performance Standards

Liners systems which have been selected for the landfills and surface impoundments are:

- compatible and durable in the presence of waste fluids (leachate) to be contained

- have low permeability over extended periods of time

- reliable and have a low risk of failure and relative ease of installation quality control, repair and maintenance.

The following design elements address the above considerations:

- all lining, cap and piping outside liners will be high density polyethylene (HDPE), the lining material most resistant to the widest group of chemical characteristics impacting liner integrity.

- a three-liner system will be used, consisting of two layers of HDPE, above a three foot thick recompacted clay liner.

- all seams and HDPE pipe connections will be heat welded, to provide as monolithic a system as possible.

- a comprehensive quality control program for soil components (clay) and the liner material (HDPE) will be followed during landfill and surface impoundments construction (refer to Quality Control Plans and Project Specifications for further information).

In general, wastes will only be accepted for direct land disposal which are known to be compatible with the liner, based on previous compatibility test data. Any new candidate wastes where compatibility data or known waste properties do not clearly indicate compatibility will either be:

- subject to additional testing for liner compatibility, or

- treated in the stabilization/fixation process to change the waste characteristics to those suitable for land disposal.

C-11b Testing of On-site Soil to Determine its Use as a Liner

On-site material, suitable for use as a liner and meeting impermeability standards ($<1.0 \times 10^{-7}$ cm/sec) will be used as the third liner (below the two synthetic liners). Previous testing of this material clearly indicates its suitability for use as an impermeable lining material. Since it is the third liner, this material will have no direct contact with the wastes themselves.

Section D-6, Landfills, discusses the strength of the soil liner and provides testing results and calculations showing the suitability of this soil as a liner. Actual construction of the liner material will be conducted following rigid quality control practices, which will include compaction and permeability testing. Refer to the Quality Control Documents and Project Specifications for further information.

A summary of the Liner Compatibility Testing is given in Table C-14.

C-12 Testing Program for Closure of the Facility [40 CRR 264.178 264.197 and 264.228]

C-12a Procedures for Assuring Decontamination of Containers

Hazardous wastes are removed from their containers by either pumping or pouring. All wastes that can be removed by using one of these two methods are removed till there is less than 2.5 centimeters (one inch) of residue remaining on the bottom of the container or inner liner. These containers are no longer considered a hazardous waste and are therefore not subject to regulation under 40 CFR 261 through 265, however, they will still be carefully managed. These containers will either be returned to the original generator or crushed and buried on-site as a hazardous waste.

C-12b Procedures for Assuring Decontamination of the Proposed Storage Facility at Closure

At closure, each of the container storage area bays will be decontaminated using a series of solvent washes followed by clean water rinses. The wastewater generated from this decontamination procedure will be collected in each of the respective containment system sumps. Samples representative of the rinsings in each storage bay will be collected from the sumps using Coliwasas. Wastewaters will be managed as hazardous waste in remaining on-site facilities.

APPENDIX C.1

TEST METHODS FOR
SELECTED ANALYTICAL PARAMETERS

BAEYER TEST

Aqueous Potassium Permanganate

An alkene or an alkyne is oxidized by potassium permanganate. The characteristic color of the permanganate ion disappears as it is consumed and this serves as an indication that a reaction has taken place.

$$3\text{-}\overset{|}{C}{=}\overset{|}{C}\text{-} + 2\underset{\text{(PURPLE)}}{MnO_4^{\ominus}} + 4H_2O \longrightarrow 3\text{-}\underset{OH}{\overset{|}{C}}\text{-}\underset{OH}{\overset{|}{C}}\text{-} + 2\underset{\text{(BROWN)}}{MnO_2} + 2OH^{\ominus}$$

A solution of 0.5 milliliter (ml) of liquid to be tested or 0.1 gram (g) of a solid to be tested, dissolved in 1 ml of ethanol or acetone, is placed in a test tube. A 2 percent potassium permanganate solution is added dropwise, and the test tube shaken after each addition. A positive test is indicated by the rapid decolorization of the permanganate solution. If a solvent is used, it is advisable to run a blank by adding the permanganate solution to 1 ml of the solvent.

Other substances that also are easily oxidized give a positive Baeyer test. Aldehydes, some alcohols, and certain polyfunctional compounds respond. Therefore, a positive Baeyer test merely indicates the presence of an easily oxidizable group.

HALIDE TEST

When it has been established that the organic compound contains a halogen, the specific structural type can be ascertained by appropriate test. That is, a distinction can be made between primary, secondary, tertiary or allyl or benzyl, and aryl or vinyl halides. The test is based on the reaction of the halide with silver nitrate solution. An alcoholic solution of the reagent is employed so that a homogeneous reaction mixture is obtained.

Aryl and vinyl halides do not react because of the difficulty of displacing the halogen in such systems. The other types of halides do react with silver nitrate and the corresponding insoluble silver halide is formed. The distinction is made from the observed rate of raction. A tertiary, allyl or benzyl type halide reacts most rapidly, and a precipitate of the silver halide is observed almost instantaneously. A secondary aliphatic halide reacts more slowly, and the precipitate does not appear until after the elapse of one to five minutes. A primary halide reacts very slowly, and the reaction mixture must be heated in order for the silver halide to precipitate out.

A drop or a few milligrams of the test compound is added to 2 ml of a 2 percent solution of silver nitrate in methanol or ethanol. If no precipitate is formed immediately, the solution is heated to boiling in a hot water bath. The absence of a precipitate after this last treatment indicates an unreactive halide.

The interpretation of the results of this test must be done carefully. For example, an aryl halide having one or more meta-directing groups ortho or para to the halogen may also react with silver nitrate solution. Thus, if it is later determined that a nitro group, for instance, is present in an aromatic compound that also contains reactive halogen, it is quite possible that the substance is an ortho or para substituted nitroaryl halide. Furthermore, a compound may contain two halogen atoms per molecule, one of which is tertiary and the other, primary. The rapid reaction of the tertiary halide will obscure the presence of the primary halide function.

AMERICAN NATIONAL STANDARD ASTM D 891 - 59 (Reapproved 1976)[1]

Standard Test Methods for SPECIFIC GRAVITY OF INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS[1]

This Standard is issued under the fixed designation D 891; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

These methods have been approved for use by agencies of the Department of Defense to replace Method 4183 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

This method has been replaced by Committee D-16 in favor of Method D 3505 and will be withdrawn from Part 29 in April 1981. Also, this method is now under the jurisdiction of Committee E-15 and will appear in Part 30.

NOTE Editorial changes were made throughout in October 1976.

1. Scope

1.1 These methods[2] cover the determination of the specific gravity (Note 1) of industrial aromatic hydrocarbons and related materials. They define suitable apparatus and procedures and furnish details underlying the interpretation of test data and the selection of numerical limits for agreement among interested persons and agencies.

NOTE—As defined in 2.1.

1.2 Four methods are covered as follows:

1.2.1 *Method A*, specific gravity by means of a specific gravity balance.

1.2.2 *Method B*, specific gravity by means of a hydrometer.

1.2.3 *Method C*, specific gravity by means of a pycnometer.

1.2.4 *Method D*, specific gravity by means of a vacuum pycnometer.

NOTE 3—In referee problems, the pycnometer method (Method C) shall be used.

2. Definitions[3]

2.1 *specific gravity*—the ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated as follows:

2.1.1 When the temperatures of the material and of the water are the *same*:

Specific gravity, x/x C

where x is the temperature of the material and the water.

2.1.2 When the temperature of the material and of the water are *not the same:*

Specific gravity, x/y C

where x is the temperature of the material and y is the temperature of the water.

2.2 *absolute specific gravity*—the ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water (Note

[1] These methods are under the jurisdiction of ASTM Committee E-15 on Industrial Chemicals and is the direct responsibility of Subcommittee E15.23 on Physical Properties.

Current edition accepted Sept. 10, 1959. Originally issued 1946. Replaces D 891 - 51.

[2] For information concerning the development of these methods, see the following references:

ASTM Definitions D 16, Terms Relating to Paint, Varnish, Lacquer, and Related Products, which appears in the *Annual Book of ASTM Standards*, Part 29; ASTM Method D 287, Test for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method), see *Annual Book of ASTM Standards*, Part 23; ASTM Definitions E 12, Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases, see *Annual Book of ASTM Standards*, Parts 14 and 41.

"Testing of Hydrometers," *Circular No. 16*, Nat. Bureau Standards. "Standard Density and Volumetric Tables," *Circular No. 19*, NBSCA, Nat. Bureau Standards.

"Methods of the Chemists of the U.S. Steel Corp. for the Sampling and Analysis of Coal, Coke, and By-Products," Carnegie Steel Co., Bureau of Technical Instruction, Pittsburgh, Pa., 1929.

Griffin, R. C., *Technical Methods of Analysis*, McGraw-Hill Book Co., Inc., New York, N. Y., 1921.

[3] These definitions are in compliance with Definitions E 12.

4) at a stated temperature. It shall be stated as follows:

2.2.1 When the temperatures of the material and of the water are the *same*:

Absolute specific gravity, x/x C

where x is the temperature of the material and the water.

2.2.2 When the temperatures of the material and of the water are *not the same*:

Absolute specific gravity, x/y C

where x is the temperature of the material and y is the temperature of the water.

NOTE 4—Gas-free distilled water is distilled water which has been boiled to eliminate dissolved gases.

3. Test Temperatures

3.1 Material specifications often specify different temperatures at which specific gravity shall be measured (Note 5), for example:

Specific gravity at 15.56/15.56 C,
Specific gravity at 20/20 C,
Specific gravity at 25/25 C, or
Specific gravity at 60/60 F

Where precision is desired, it is necessary to determine the specific gravity at the temperature prescribed in the specifications for the material to be tested and to use instruments that have been calibrated and standardized at the specified temperature.

NOTE 5—For the significance and interpretation of expressions such as "specific gravity at 25/15.56 C" see 14.2.

3.2 For some of the liquids, an agreement may be reached as to the coefficient of expansion of the product. In such cases the specific gravity may be changed from one temperature basis to another as described in 14.3.

METHOD A—SPECIFIC GRAVITY BY MEANS OF A SPECIFIC GRAVITY BALANCE

4. Apparatus

4.1 *Balance*—Westphal or equivalent balance, capable of determining specific gravity to the fourth decimal place, and equipped with a plummet for use at 15.56 C.

4.2 *Cylinder*—The cylinder may be of metal or of clear glass, with a lip on the rim for convenience in pouring. A convenient size of cylinder is approximately 1½ in. (38.1 mm) in diameter and 5 in. (127 mm) in height.

4.3 *Thermometer*—An ASTM Gravity Thermometer having a range from −20 to +102 C and conforming to the requirements for Thermometer 12C as prescribed in ASTM Specifications E 1, for ASTM Thermometers.[4, 5]

4.4 *Water Bath*—A water bath capable of maintaining a temperature of 15.56 ± 0.05 C during the test.

5. Procedure

5.1 Cool the sample in the original closed container to the temperature specified for the test. The thermometer, plummet, and cylinder should be at about the same temperature. Take care to prevent the temperature of the sample from changing appreciably during the time necessary to complete the test. This may be done by placing the test cylinder in the water bath maintained at the necessary temperature during the time of the test. Sufficient time shall be allowed to permit the plummet, cylinder, and thermometer to reach the specified temperature. In cases of dispute it is recommended that the temperature of the sample be determined both before and after the reading is taken.

5.2 Suspend the plummet from the arm of the balance and adjust the balance to equilibrium in air so that the reading is zero. Pour freshly boiled and cooled distilled water into the cylinder. Place the cylinder in the water bath and arrange so that the plummet is immersed in the distilled water. Be sure that the plummet is free of air bubbles, is completely submerged, and does not touch the sides or bottom of the cylinder containing the sample. Stir the contents of the cylinder and, when the temperature is 15.56 ± 0.05 C, take the balance reading. Wash the plummet and cylinder with alcohol and ether to clean and dry. Pour enough of the previously cooled sample into the cylinder to cause the suspension wire to be immersed to the same point as before and repeat the above procedure to ob-

[4] *Annual Book of ASTM Standards*, Part 25.
[5] *Annual Book of ASTM Standards*, Part 44.

tain the reading when the plummet is immersed in the sample at 15.56 C.

6. Calculation

6.1 Calculate the specific gravity as follows:

$$\text{Specific gravity at } 15.56/15.56\text{ C} = A/B$$

where:

A = reading obtained with sample, and
B = reading obtained with water.

METHOD B—SPECIFIC GRAVITY BY MEANS OF A HYDROMETER

7. Apparatus

7.1 *Hydrometer*—The hydrometers to be used shall be those specified in ASTM Specification E 100, for ASTM Hydrometers,[4,5] as follows:

Nominal Specific Gravity Range	Hydrometer
0.850 to 0.900	86H
0.900 to 0.950	87H
0.950 to 1.000	88H

NOTE 6—Corresponding hydrometers calibrated with the API scale may be used and the API reading converted to specific gravity with Table 3 of D 1250, Petroleum Measurement Tables.[3] In case of dispute the 86H, 87H, and 88H hydrometers shall be controlling.

7.2 *Hydrometer Cylinder*—The vessel in which the sample for the gravity test is confined shall be made of clear glass and shall be cylindrical in shape. For convenience in pouring, it may have a lip on the rim. The inside diameter shall be at least 25.4 mm (1.0 in.) greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that after equilibrium has been reached, the lowest point on the hydrometer will be at least 25 mm (1 in.) off the bottom of the cylinder.

7.3 *Thermometer*—See 4.3.

7.4 *Water Bath*—See 4.4.

8. Procedure

8.1 Cool the sample in the original container to about 14 C. Rinse each piece of equipment with a portion of the sample and discard the rinse liquid. Pour the sample into the clean hydrometer cylinder without splashing, so as to avoid formation of air bubbles. Remove any air bubbles adhering to the surface by touching them with a piece of clean filter paper. Select a location that is free from air currents. Place the cylinder vertically in the water bath and let the temperature of the sample reach 15.56 ± 0.05 C as follows: Stir the contents of the cylinder, being careful to avoid formation of air bubbles. When the temperature of the sample is 15.3 C, slowly and carefully lower the hydrometer into the sample to a level two smallest scale divisions below that at which it will float and then release the hydrometer. After it has come to rest and floats freely away from the walls of the cylinder, read the gravity as the point at which the surface of the sample apparently cuts the hydrometer scale. When the temperature is 15.56 C, make this observation by placing the eye slightly below the level of the liquid and slowly raise the eye until the surface of the sample first seen as a distorted ellipse seems to become a straight line cutting the hydrometer scale. Determine the temperature of the sample just before and also, for referee tests, just after reading the hydrometer.

METHOD C—SPECIFIC GRAVITY BY MEANS OF A PYCNOMETER[6]

9. Apparatus

9.1 *Pycnometer*—A pycnometer of 25-ml capacity with a ground-glass stopper having a capillary opening, a chamber to provide for expansion up to room temperature, and a cap to prevent evaporation.

9.2 *Water Bath*—A water bath capable of maintaining a temperature of 15.56 ± 0.05 C, or other required temperature, during the test.

9.3 *Thermometer*—An ASTM Low Softening Point Thermometer having a range from −2 to +80 C and conforming to the requirements for Thermometer 15C as prescribed in Specifications E 1.

9.4 *Analytical Balance*—A balance having a sensitivity of between 2 and 3 scale divisions displacement effected by an excess weight of 1 mg when carrying a load of between 15 and 50 g in each pan.

[6] For a high degree of accuracy, the following paper discusses an apparatus and method of much merit:

Lipkin and Associates, "Pycnometer for Volatile Liquids," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 36, Jan. 15, 1944, pp. 55-58.

The Bingham and other pycnometers employing capillary tubing use the same principle as described in the paper.

9.5 *Analytical Weights*—Class S weights, as certified by the National Bureau of Standards, or equivalent weights.

10. Procedure

10.1 Clean the pycnometer by filling it with a saturated solution of chromic acid in sulfuric acid (H_2SO_4, sp gr 1.84), allowing it to stand for a few hours, emptying, and rinsing well with distilled water. Fill the pycnometer with freshly boiled distilled water which has been cooled to 12 to 14 C. Place it in the water bath maintained at 15.56 ± 0.05 C until the pycnometer and its contents are at a constant volume at 15.56 C. After immersion in the bath for at least 30 min, adjust the level of liquid to the proper point on the pycnometer, put the stopper in place, remove from the bath, wipe dry, and weigh. Empty the pycnometer, rinse successively with alcohol and ether, remove the ether vapor, immerse in the bath, and bring to 15.56 C as was done before. After immersion at 15.56 C for at least 30 min, put the stopper in place, remove from the bath, wipe dry, and weigh. Subtract the weight of the empty pycnometer from the weight when filled with water in order to get the weight of the contained water at 15.56 C in air. Call this difference W. Cool the sample to 12 to 14 C, fill the pycnometer with it, immerse in the bath and bring to 15.56 C as was done before. After immersion at 15.56 C for at least 30 min, adjust the liquid level, put the stopper in place, remove from the bath, wipe dry, and weigh. Subtract the weight of the empty pycnometer from the weight when filled with sample in order to obtain the weight of the contained sample at 15.56 C. Call this difference S.

11. Calculation

11.1 Calculate the specific gravity at 15.56/15.56 C (in air) as follows:

$$\text{Specific gravity at } 15.56/15.56 \text{ C} = S/W$$

METHOD D—SPECIFIC GRAVITY BY MEANS OF A VACUUM PYCNOMETER

NOTE 7—This method is suitable for use where an agreement has been reached concerning the magnitude of the coefficient of cubical expansion of the material at different temperatures.

12. Apparatus

12.1 *Pycnometer*—A vacuum-wall pycnometer, 25 ml in capacity.

12.2 *Thermometer*—See 9.3.

12.3 *Analytical Balance*—See 9.4.

12.4 *Analytical Weights*—See 9.5.

13. Procedure

13.1 Using the procedure described in 13.2, determine at two different temperatures (for example, 15 and 30 C) the weights of water required to fill the pycnometer at these temperatures. Retain the data for use in calculating the weight of water required to fill the pycnometer at any particular temperature at which it is subsequently employed, as outlined in 13.2. Call this calculated weight W. Dry the pycnometer and keep it ready for use.

13.2 Transfer about 200 ml of the sample at room temperature to a clean, dry 300-ml beaker. Rinse the pycnometer with some of the sample, immerse it in the beaker, and allow it to fill. Insert the thermometer in the beaker. Let the apparatus stand several minutes to permit the sample and the instruments to reach the same temperature. Empty the pycnometer and immerse it again until it is filled with the sample. Record the temperature in degrees Celsius (x). Remove the pycnometer, insert the capillary tube stopper, and quickly wipe dry, taking care to see that the ground-glass cap joint is dried and that none of the liquid is removed from the capillary. Put the glass cap in place and weigh at once. Clean and dry the pycnometer and obtain its weight when empty. Record the weight W' of the sample required to fill the pycnometer at x degrees Celsius.

14. Calculation

14.1 Calculate the specific gravity as follows:

$$S_x = W'/W$$

$$S_o = S_x[1 + k(x - 15.56)](d_x/d_{15.56})$$

where:

S_x = specific gravity at x/x C,

S_o = specific gravity at 15.56/15.56 C,

W and W' have the values stated in 13.1 and 13.2 respectively,

x = temperature at which test was made,

k = coefficient of cubical expansion (0.00124 for industrial pure benzene and 0.00110 for industrial pure toluene),

d_x = absolute density of water at x degrees Celsius, and

$d_{15.56}$ = absolute density of water at 15.56 C (taken as 0.99901).

14.2 The expression "specific gravity at 25.0/15.56 C," for example, means the ratio of the weight in air of a given volume of material at 25.0 C to the weight in air of an equal volume of pure water at 15.56 C. This cannot be determined directly due to the fact that the volume of the pycnometer is not the same at 25 C as at 15.56 C. A simple and accurate procedure for obtaining the specific gravity of a sample at x/15.56 C consists in determining the weight in air of that amount of sample which fills the pycnometer at temperature x C, dividing this by W (the water weight in air at the same temperature x C) and correcting for the change in the water weight between x C and 15.56 C. The water weight in the pycnometer at x C is derived as follows from the data obtained in 14.1: Divide the observed water weights (in air) at each of two temperatures by the corresponding absolute densities of water and interpolate linearly between these values to find an "apparent volume" of the pycnometer at x C. This "apparent volume" multiplied by the absolute density of water at x C gives the value of W. The specific gravities at x/x C and at x/15.56 C are inversely proportional to the absolute densities of water at x C and 15.56 C. Absolute densities of water at different temperatures are tabulated in various handbooks.

14.3 If the coefficient of cubical expansion of the sample is known, its specific gravity, S_2, at x_2/y C may be calculated from the specific gravity, S_1, at x_1/y C by using the following equation:

$$S_2 = S_1[1 + k(x_1 - x_2)]$$

where k is the coefficient of cubical expansion over the proper temperature range. This relationship holds provided that the values of S_1 and S_2 are based upon water of the same reference temperature y C. By differentiation of this equation, it becomes evident that an error of 0.1 C in measuring the temperature of the sample will cause an error in the specific gravity figure approximately equal to the numerical value of the expression, $0.1\ kS_1$.

14.4 Optionally, one may calibrate the pycnometer directly at the same temperature at which it is used.

15. Precision

15.1 When Method A, C, or D is employed, different laboratories using different instruments should be able to obtain results that differ from the mean by not more than 0.0002.

15.2 The limits of precision and accuracy of any method for determining specific gravity depend upon the attention which is given to details of calibration and technique. Consideration, in general, must be given to the problems of keeping a large volume of liquid (sometimes unstirred) at a constant temperature, providing for the effects of humidity or static electricity during weighing of pycnometers, and weighing relatively large loads. When extreme accuracy is desired, instruments certified by the National Bureau of Standards shall be used and the necessary corrections applied.

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

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

Designation: D 2111 - 71 (Reapproved 1978)

Standard Test Methods for SPECIFIC GRAVITY OF HALOGENATED ORGANIC SOLVENTS AND THEIR ADMIXTURES[1]

This Standard is issued under the fixed designation D 2111; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

These methods have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These methods cover the determination of the specific gravity of halogenated organic solvents and solvent admixtures. They define suitable apparatus and procedures and furnish details underlying the interpretation of test data and the selection of numerical limits for agreement among interested persons and agencies.

1.2 Four methods are covered as follows:

1.2.1 *Method A*, specific gravity by means of a specific gravity balance.

1.2.2 *Method B*, specific gravity by means of a hydrometer.

1.2.3 *Method C*, specific gravity by means of a pycnometer.

1.2.4 *Method D*, specific gravity by means of a vacuum pycnometer.

NOTE 1—In referee problems, Methods A, C, or D may be used.

2. Definitions

2.1 *specific gravity*—the ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

2.1.1 When the temperatures of the material and of the water are the same:

Specific gravity x/x C. . . .

where x = temperature of the material and the water.

2.1.2 When the temperature of the material and of the water are not the same:

Specific gravity x/y C. . . .

where:

x = temperature of the material, and

y = temperature of the water.

2.1.3 *specific gravity, absolute*—the ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water (Note 2) at a stated temperature. It shall be stated as in 2.1.

NOTE 2—Gas-free distilled water is distilled water that has been boiled to eliminate dissolved gases.

3. Test Temperatures

3.1 Material specifications often specify different temperatures at which specific gravity shall be measured:

Specific gravity 15/4 C
Specific gravity 20/20 C
Specific gravity 25/25 C

For purposes of unity, the test temperature used throughout shall be 25/25 C.

3.2 For some of the liquids, an agreement may be reached as to the coefficient of expansion of the product. In such cases, the specific gravity may be changed from one temperature basis to another as described in Section 14.

METHOD A—SPECIFIC GRAVITY BY MEANS OF A SPECIFIC GRAVITY BALANCE

4. Apparatus

4.1 *Balance*—Westphal or equivalent, capable of determining specific gravity to the

[1] These methods are under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents.

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fourth decimal place, and equipped with a plummet for use at 25 C.

4.2 *Cylinder*—The cylinder may be of metal or of clear glass, with a lip on the rim for convenience in pouring. A convenient size of cylinder is approximately 1½ in. (38 mm) in diameter and 5 in. (127 mm) in height.

4.3 *Thermometer*—An ASTM Gravity Thermometer having a range from −20 to +102 C and conforming to the requirements for Thermometer 12C as prescribed in ASTM Specification E 1.[2]

4.4 *Water Bath*, capable of maintaining a temperature of 25.0 ± 0.5 C during the test.

5. Procedure

5.1 Cool the sample in the original closed container to the temperature specified for the test. The thermometer, plummet, and cylinder should be at about the same temperature. Take care to prevent the temperature of the sample from changing appreciably during the time necessary to complete the test. This may be done by placing the test cylinder in the water bath maintained at the necessary temperature during the time of the test. Allow sufficient time to permit the plummet, cylinder, and thermometer to reach the specified temperature. In cases of dispute, it is recommended that the temperature of the sample be determined both before and after the reading is taken.

5.2 Suspend the plummet from the arm of the balance and adjust the balance to equilibrium in air so that the reading is zero. Pour freshly boiled and cooled distilled water into the cylinder. Place the cylinder in the water bath and arrange so that the plummet is immersed in the distilled water. Be sure that the plummet is free of air bubbles, is completely submerged, and does not touch the sides or bottom of the cylinder containing the sample. Stir the contents of the cylinder and, when the temperature is 25.0 ± 0.5 C, take the balance reading. Wash the plummet and cylinder with alcohol and ether to clean and dry. Pour enough of the previously cooled sample into the cylinder to cause the suspension wire to be immersed to the same point as before, and repeat the above procedure to obtain the reading when the plummet is immersed in the sample at 25 C.

6. Calculation

6.1 Calculate the specific gravity as follows:

$$\text{Specific gravity } 25/25\text{ C} = A/B$$

where:

A = reading obtained with the sample, and
B = reading obtained with water.

Method B—Specific Gravity by Means of a Hydrometer

7. Apparatus

7.1 *Hydrometer*—The hydrometers to be used shall be those specified in ASTM Specification E 100, for ASTM Hydrometers[3] as follows:

Nominal Specific Gravity Range	ASTM Hydrometer No.
0.900 to 0.950	107H
0.950 to 1.000	108H
1.000 to 1.050	125H
1.050 to 1.100	126H
1.100 to 1.150	127H
1.150 to 1.200	128H
1.200 to 1.250	129H
1.250 to 1.300	130H
1.300 to 1.350	131H
1.350 to 1.400	132H
1.400 to 1.450	133H
1.450 to 1.500	134H
1.500 to 1.550	135H
1.550 to 1.600	136H
1.600 to 1.650	137H

7.2 *Hydrometer Cylinder*—The vessel in which the sample for the gravity test is confined shall be made of clear glass and shall be cylindrical in shape. For convenience in pouring, it may have a lip on the rim. The inside diameter shall be at least 25.4 mm (1.0 in.) greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that the length of the column of sample it contains is greater by at least 25.4 mm (1.0 in.) than the portion of the hydrometer that is immersed beneath the surface of the sample after a state of equilibrium has been reached.

7.3 *Thermometer*—See 4.3.

7.4 *Water Bath*—See 4.4.

8. Procedure

8.1 Cool the sample in the original container to about 24 C. Rinse each piece of equipment with a portion of the sample. Pour the sample

[2] *Annual Book of ASTM Standards*, Part 25 and 44.

into the clean hydrometer cylinder without splashing, so as to avoid formation of air bubbles. Remove any air bubbles adhering to the surface by touching them with a piece of clean filter paper. Select a location that is free of air currents. Place the cylinder vertically in the water bath and let the temperature of the sample reach 25.0 ± 0.5 C as follows: Stir the contents of the cylinder, being careful to avoid formation of air bubbles. When the temperature of the sample is 24.5 C, slowly and carefully lower the hydrometer into the sample to a level two smallest scale divisions below that at which it will float and then release the hydrometer. After it has come to rest and floats freely away from the walls of the cylinder, read the gravity as the point at which the surface of the sample apparently cuts the hydrometer scale.

8.2 When the temperature is 25.0 C, make this observation by placing the eye slightly below the level of the liquid and slowly raise the eye until the surface of the sample first seen as a distorted ellipse seems to become a straight line cutting the hydrometer scale. Determine the temperature of the sample just before and also, for referee tests, just after reading the hydrometer.

Method C—Specific Gravity by Means of a Pycnometer

9. Apparatus

9.1 *Pycnometer*, 25-ml capacity with a ground-glass stopper having a capillary opening, a chamber to provide for expansion up to room temperature, and a cap to prevent evaporation.

9.2 *Water Bath*, capable of maintaining a temperature of 25.0 ± 0.5 C, or other required temperature, during the test.

9.3 *Thermometer*—An ASTM Low Softening Point Thermometer having a range from −2 to +80 C and conforming to the requirements for thermometer 15C as prescribed in Specifications E 1.[4]

9.4 *Analytical Balance*, having a sensitivity of between 2 and 3 scale divisions displacement effected by an excess weight of 1 mg when carrying a load of between 15 and 50 g in each pan.

9.5 *Analytical Weights*, Class S, as certified by the National Bureau of Standards, or equivalent weights.

10. Procedure

10.1 Clean the pycnometer by filling it with a saturated solution of chromic acid in concentrated sulfuric acid (H_2SO_4, sp gr 1.84), allowing it to stand for a few hours, emptying, and rinsing well with distilled water. Fill the pycnometer with freshly boiled distilled water that has been cooled to 22 to 24 C. Place it in the water bath maintained at 25.0 ± 0.5 C until the pycnometer and its contents are at a constant volume at 25 C.

10.2 After immersion in the bath for at least 30 min, adjust the level of liquid to the proper point on the pycnometer, put the stopper in place, remove from the bath, wipe dry, and weigh. Empty the pycnometer, rinse successively with alcohol and ether, remove the ether vapor, immerse in the bath and bring to 25.0 C as was done before. After immersion at 25.0 C for at least 30 min, put the stopper in place, remove from the bath, wipe dry, and weigh. Subtract the weight of the empty pycnometer from the weight when filled with water in order to get the weight of the contained water at 25.0 C in air. Call this difference W.

10.3 Cool the sample to 22 to 24 C, fill the pycnometer with it, immerse in the bath, and bring to 25.0 C as was done before. After immersion at 25.0 C for at least 30 min, adjust the liquid level, put the stopper in place, remove from the bath, wipe dry, and weigh. Subtract the weight of the empty pycnometer from the weight when filled with sample in order to obtain the weight of the contained sample at 25.0 C. Call this difference S.

11. Calculation

11.1 Calculate the specific gravity at 25/25 C (in air) as follows:

$$\text{Specific gravity } 25/25\text{ C} = S/W$$

Method D—Specific Gravity by Means of a Vacuum Pycnometer

12. Apparatus

12.1 *Pycnometer*—A vacuum-wall pycnometer, 25 ml in capacity.

12.2 *Thermometer*—See 9.3.

12.3 *Analytical Balance*—See 9.4.

12.4 *Analytical Weights*—See 9.5.

13. Procedure

13.1 Using the procedure described in 13.2, determine at two different temperatures (for example, 15 and 30 C) the weights of water required to fill the pycnometer at these temperatures. Retain the data for use in calculating the weight of water required to fill the pycnometer at any particular temperature at which it is subsequently employed, as outlined in 14.2. Call this calculated weight W. Dry the pycnometer and keep it ready for use.

13.2 Transfer about 200 ml of the sample at room temperature to a clean, dry 300-ml beaker. Rinse the pycnometer with some of the sample, immerse it in the beaker, and allow it to fill. Insert the thermometer in the beaker. Let the apparatus stand several minutes to permit the sample and the instruments to reach the same temperature. Empty the pycnometer and immerse it again until it is filled with the sample. Record the temperature in degrees Celsius, x. Remove the pycnometer, insert the capillary tube stopper, and quickly wipe dry, taking care to see that the ground-glass cap joint is dried and that none of the liquid is removed from the capillary. Put the glass cap in place and weigh at once. Clean and dry the pycnometer and obtain its weight when empty. Record the weight W' of the sample required to fill the pycnometer at x C.

14. Calculation

14.1 Calculate the specific gravity as follows:

$$S_x = W'/W$$

$$S_x = S_x\,[1 + k\,(x - 25.0)]\,(d_x/d_{25.0})$$

where:

S_x = specific gravity x/x C,
S_x = specific gravity 25/25 C,
W, W' = values stated in 13.1 and 13.2, respectively,
x = temperature at which the test was made,
k = coefficient of cubical expansion (0.00117 at 0 to 40 C for trichloroethylene, 0.00102 at 0 to 25 C for perchloroethylene, 0.00125 at 0 to 30 C for 1,1,1-trichloroethane, 0.00137 at 0 to 40 C for methylene chloride, and 0.000927 at 0 to 30 C for fluorocarbon-113)
d_x = absolute density of water at xC, and
d_{25} = absolute density of water at 25 C (taken as 0.997044).

14.2 Material specifications often specify different temperatures at which specific gravity shall be measured. In order to convert to any selected temperature, the coefficient of cubical expansion of 0.00117 for trichloroethylene and 0.00102 for perchloroethylene are employed. In addition, the absolute density of water at the desired temperature is taken. The absolute densities of water at different temperatures are tabulated in various handbooks. In converting to any desired temperature basis the following equation is used:

$$\text{Specific gravity } T_3/T_4\ C = \frac{\text{specific gravity } T_1/T_2}{[1 + k(T_3 - T_1)]\,\left(\dfrac{d_{H_2O} \text{ at } T_4}{d_{H_2O} \text{ at } T_2}\right)}$$

where:

T_1/T_2 = original temperature conditions,
T_3/T_4 = new temperature conditions,
k = coefficient of cubical expansion (0.00117 at 0 to 40 C for trichloroethylene, 0.00102 at 0 to 25 C for perchloroethylene, 0.00125 at 0 to 30 C for 1,1,1-trichloroethane, 0.00137 at 0 to 40 C for methylene choride, and 0.000927 at 0 to 30 C for fluorocarbon-113)
d_{H_2O} = absolute density of water at the specified temperature.

NOTE 3: *Example*—Assume a specific gravity of 1.4550 at 25/25 C for trichloroethylene and this is to be converted to 15/4 C and 20/20 C.

Specific gravity 15/4 C
= 1.4550/[1 + 0.00117(15 − 25)] (0.999973/0.997044)
= 1.4550/[0.9883] 1.00293
= 1.4679

where 0.999973/0.997044 = ratio of the absolute density of water at 4 C compared to the absolute density of water at 25 C.

Specific gravity 20/20 C
= 1.4550/[1 + 0.00117 (20 − 25)] (0.998203/0.997044)
= 1.4550/[0.99415] 1.00116
= 1.4619

where 0.998203/0.997044 = ratio of the absolute density of water at 20 C compared to the absolute density of water at 25 C.

NOTE 4—Figures for absolute density of water are taken from the *Handbook of Chemistry and Physics*, Forty-ninth edition (1968–1969), published by The Chemical Rubber Co.

15. Precision

15.1 When Method A, C, or D is employed, different laboratories using different instruments should be able to obtain results that differ from the means by not more than 0.0002.

15.2 The limits of precision and accuracy of any method for determining specific gravity depend upon the attention that is given to details of calibration and technique. Consideration, in general, must be given to the problems of keeping a large volume of liquid (sometimes unstirred) at a constant temperature, providing for the effects of humidity or static electricity during weighing of pycnometers, and weighing relatively large loads. When extreme accuracy is desired, instruments certified by the National Bureau of Standards shall be used and the necessary corrections applied.

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

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

Standard Test Method for WATER USING KARL FISCHER REAGENT[1]

is Standard is issued under the fixed designation E 203; the number immediately following the designation indicates e year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the ar of last reapproval.

is method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

. Scope

1.1 This method is intended as a general guide for the application of the Karl Fischer reagent method for determining free water and water of hydration in most solid or liquid organic and inorganic compounds. Samples that are gaseous at room temperature are not covered (see Appendix A4). By proper choice of sample size, Karl Fischer reagent concentration, and apparatus, the method is suitable for the measurement of water over a wide concentration range, that is, parts per million to pure water. Both visual and electrometric methods are described for ascertaining the end point.

1.2 A list of existing ASTM Karl Fischer reagent methods, their applications to various products, and the sponsoring committees is given in Appendix A3.

2. Summary of Method

2.1 The sample, containing a maximum of 300 mg of water, is dissolved or dispersed in a suitable liquid and titrated with Karl Fischer reagent, which is a mixture of iodine, sulfur dioxide, pyridine, and methanol or glycol ether. As long as any water is present, the iodine is reduced to colorless hydrogen iodide. The end point is the first appearance of free iodine, determined either visually or electrometrically. In some cases it may be desirable to add excess Karl Fischer reagent, and then back-titrate with methanol containing a known concentration of water.

2.2 Fundamental equations are as follows:

$$C_5H_5N \cdot I_2 + C_5H_5N \cdot SO_2 + C_5H_5N + H_2O \rightarrow 2C_5H_5N \cdot HI + C_5H_5N \cdot SO_3$$

$$C_5H_5N \cdot SO_3 + ROH \rightarrow C_5H_5N \cdot HSO_4R$$

NOTE 1: **Caution**—Karl Fischer reagent contains four toxic compounds, namely, iodine, sulfur dioxide, pyridine, and methanol or glycol ether. The reagent should be dispensed in a well-ventilated area. Care must be exercised to avoid unnecessary inhalation of the reagent or direct contact of the reagent with the skin. Following accidental spillage, wash with large quantities of water.

3. Applications

3.1 Applications can be subdivided into two sections: (*1*) organic and inorganic compounds in which water may be determined directly, and (*2*) compounds in which water cannot be determined directly, but in which interferences may be eliminated by suitable chemical reactions or modifications of the procedure. Further discussion of interferences is included in Appendix A2.

3.2 Water can be determined directly in the presence of the following types of compounds:

ORGANIC COMPOUNDS

Acetals	Halides
Acids (Note 2)	Hydrocarbons (saturated and unsaturated)
Acyl halides	Ketones, stable (Note 5)
Alcohols	Nitriles
Aldehydes, stable (Note 3)	Orthoesters
Amides	Peroxides (hydro, dialkyl)
Amines, weak (Note 4)	Sulfides
Anhydrides	Thiocyanates
Disulfides	Thioesters
Esters	
Ethers	

INORGANIC COMPOUNDS

Acids (Note 6)	Cupric oxide
Acid oxides (Note 7)	Desiccants
Aluminum oxides	Hydrazine sulfate
Anhydrides	Salts of organic and inorganic acids (Note 7)
Barium dioxide	
Calcium carbonate	

[1] This method is under the jurisdiction of ASTM Committee E-15 on Industrial Chemicals.

Current edition approved April 25, 1975. Published June 1975. Originally published as E 203 - 62 T. Last previous edition E 203 - 64 (1971).

NOTE 2—Some acids, such as formic, acetic, and adipic acid, are slowly esterified. For high accuracy, use 30 to 50 percent pyridine in methanol as the solvent.

NOTE 3—Examples of stable aldehydes are formaldehyde, sugars, chloral, etc. Formaldehyde polymers contain water as methylol groups. This combined water is not titrated. Addition of an excess of $NaOCH_3$ in methanol permits release and titration of this combined water, after approximate neutralization of excess base with acetic acid (see Note 10).

NOTE 4—Weak amines are considered to be those with K_b value $< 2.4 \times 10^{-5}$.

NOTE 5—Examples of stable ketones are diisopropyl ketone, camphor, benzophenone, benzil, dibenzolacetone, etc.

NOTE 6—Sulfuric acid up to a concentration of 92 percent may be titrated directly; for higher concentrations see Note 14.

NOTE 7—Compounds subject to oxidation-reduction reactions in an iodine - iodide system interfere.

3.3 A number of substances and classes of compounds interfere in the determination of water by this titrimetric method, complete descriptions of which are found in the literature (1).[2] This interference is associated with condensation or oxidation-reduction reactions.

3.4 Interferences of many classes of compounds can be eliminated by chemical reactions to form inert compounds prior to titration. The following are in this category:

Aldehydes and ketones, active (Note 8)
Amines, strong (Note 9)
Ammonia (Note 10)
Ferric salts (Note 11)
Hydrazine derivatives (Note 10)
Hydroxylamine salts (Note 12)
Mercaptans (Note 13)
Sodium methylate (Note 10)
Sulfuric acid (Note 14)
Thioacids (Note 13)
Thiourea (Note 13)

NOTE 8—This interference may be reduced by use of pyridine rather than methanol as solvent for the sample or by the use of Karl Fischer reagent and solvent prepared with ethylene glycol monomethyl ether in place of methanol. The cyanhydrin reaction may be used to eliminate the interference (1).

NOTE 9—Strong amines are considered to be those with K_b value $> 2.4 \times 10^{-5}$. Use salicylic acid-methanol solution (Section 5). Glacial acetic acid is applicable in certain cases.

NOTE 10—Addition of acetic acid eliminates the interference.

NOTE 11—Ferric fluoride does not interfere. Reaction with 8-hydroxyquinoline is reported to eliminate this interference (7).

NOTE 12—Add 1 *M* SO_2 in 1+1 pyridine-methanol or spent Karl Fischer reagent.

NOTE 13—Olefin addition reaction eliminates interferences (1). Oxidation with neutral iodine solution eliminates the interference of mercaptans (8).

NOTE 14—Sulfuric acid, above 92 percent. Add the sample (10 g) to a large excess of pyridine (35 ml), swirl to dissolve precipitate, and titrate. Addition of 8 ml of 1+1 pyridine - dioxane/1 g of sample also is satisfactory, maintaining a homogeneous solution throughout the titration.

3.5 Many materials react stoichiometrically with Karl Fischer reagent. When their concentration is known, suitable corrections can be applied. A list of such materials is given in Appendix A2.

4. Apparatus

4.1 A suggested assembly of the apparatus, to provide a closed system during titration, is shown in Appendix A1, Fig. A1.

4.2 This equipment, without the end point detector, may be used for visual titration.

5. Reagents

5.1 Reagent grade chemicals or equivalent as specified in ASTM Methods E 200, Preparation, Standardization and Storage of Standard Solutions for Chemical Analysis,[3] shall be used in all tests.

5.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to ASTM Specification D 1193, for Reagent Water.[4]

5.3 *Karl Fischer Reagent*—The Karl Fischer reagent may either be prepared in the laboratory or purchased. Two types of reagent are commonly used. Directions for preparing these (**Caution**, see Note 15) and diluting if necessary, along with commercial sources of supply, are as follows:

NOTE 15: **Caution**—Follow standard precautions for handling toxic gases in preparing reagents (*1*) or (*2*) below.

5.3.1 *Karl Fischer Reagent (Ethylene Glycol Monomethyl Ether Solution, 1 ml = 6 mg H_2O)* (2)—For each liter of solution, dissolve 133 ± 1 g of iodine in 425 ± 5 ml of pyridine in a dry glassstoppered bottle. Add 425 ± 5 ml of ethylene glycol monomethyl ether. Cool to below 4 C in an ice bath. Bubble 102 to 105 g of gaseous sulfur dioxide (SO_2) into the cooled mixture. Determine the amount of

[2] The boldface numbers in parentheses refer to the list of references appended at the end of this method.

[3] *Annual Book of ASTM Standards*, Parts 30 and 41.

[4] *Annual Book of ASTM Standards*, Part 31.

SO_2 added by the change in weight of the SO_2 cylinder or the increase in volume (about 70 ml) of the reagent mixture. Alternatively, add about 70 ml of freshly drawn liquid SO_2 in small increments. Mix well and set aside for at least 12 h before using.

5.3.2 *Karl Fischer Reagent (Methanol Solution, 1 ml = 6 mg H_2O)*—For each liter of solution, dissolve 133 ± 1 g of iodine in 425 ± 5 ml of pyridine in a dry, glass-stoppered bottle. Add 425 ± 5 ml of methanol. Cool the mixture in an ice bath to below 4 C. Bubble 102 to 105 g of gaseous sulfur dioxide (SO_2) into the cooled mixture. Determine the amount of SO_2 added by the change in weight of the SO_2 cylinder or the increase in volume (about 70 ml) of the reagent mixture. Alternatively, add about 70 ml of freshly drawn liquid SO_2 in small increments. Mix well and set aside for at least 12 h before using.

5.3.3 *Karl Fischer Reagent (Ethylene Glycol Monomethyl Ether Solution, stabilized, 1 ml = 6 mg H_2O).*[5]

5.3.4 *Karl Fischer Reagent (Methanol Solution, Stabilized, 1 ml = 5 mg H_2O).*[6]

5.3.5 *Karl Fischer Reagent, Dilute*—Prepare more dilute solutions of the Karl Fischer reagent by diluting with the proper solvent as follows:

Desired Strength mg H_2O/ml	Liters of Diluent to Add/liter of 6 mg/ml KF Reagent
3	0.85
2	1.6
1	3.2
0.5	5.7

These dilute solutions cannot be prepared by simple proportion, since water added with the diluent must be accounted for. The volumes to add, indicated above, are calculated assuming the diluent contains 0.05 percent water.

5.4 *Methanol, Standard (1 ml = 1 mg H_2O)*[7]—This solution can be stored conveniently in a bottle with rubber cap and portions removed with a hypodermic syringe.

5.5 *Sodium Tartrate Dihydrate*—Grind certified material (water content 15.61 to 15.71 percent) to a fine powder (preferably overnight in a sealed ball mill) and store the ground material in a stoppered bottle. If doubt exists as to its water content, dry a 2 to 3-g sample in an oven at 155 ± 5 C to constant weight (minimum 4 h). (See Note 16.)

5.6 *Solvents:*

5.6.1 *Acetic Acid*, glacial.

5.6.2 *Ethylene Glycol Monomethyl Ether*, maximum 0.1 percent water.

5.6.3 *Methanol*, maximum 0.15 percent water,[8] in accordance with Specification D 1152, for Methyl Alcohol (Methanol).[9]

5.6.4 *Pyridine*, maximum 0.1 percent water.

5.7 *Solvents, Mixed*—In addition to the single solvents (5.6), the following mixed solvents are useful for dissolving various samples:

5.7.1 *Methanol - Chloroform (1+3)*—Mix 1 volume of methanol with 3 volumes of chloroform. Use for liquid petroleum products.

5.7.2 *Methanol - Salicylic Acid*—Dissolve 150 g of salicylic acid in 1 liter of methanol. Use for amines.

5.7.3 *Pyridine - Ethylene Glycol (1+4)*—Mix 1 volume of pyridine with 4 volumes of ethylene glycol. Use for compounds containing carbonyl groups.

5.7.4 *Pyridine - Methanol (1 + 4)*—Mix 1 volume of pyridine with 4 volumes of methanol. Use for organic acids.

5.8 *Sulfur Dioxide*, anhydrous grade.

6. Drying of Solvents

6.1 If it is necessary to prepare dry solvents in the laboratory, the following three methods can be used:

6.1.1 Distillation of methanol from magnesium, to reduce the moisture content to 0.005 percent, according to Section 6 of ASTM Method D 1744, Test for Water in Liquid Petroleum Products by Karl Fischer Reagent.[10]

6.1.2 Azeotropic distillation using benzene, to reduce the moisture to 0.05 percent. Add 1 volume of benzene to 19 volumes of pyridine, ethylene glycol monomethyl ether, or mixtures thereof, and distill. Discard the first

[5] Fisher Scientific Co. Catalog No. So-K-3 has been found satisfactory.

[6] Mallinckrodt Catalog No. 5651, has been found satisfactory.

[7] Fisher Scientific Co. Catalog No. So-W-2 and Hartman-Leddon Co. Catalog No. 1849 have been found satisfactory.

[8] Other acceptable grades are Mallinckrodt Catalog No. 3017, maximum 0.01 percent water, or Fisher Scientific Co. Catalog No. A-412, maximum 0.1 percent water.

[9] *Annual Book of ASTM Standards*, Part 29.

[10] *Annual Book of ASTM Standards*, Part 24.

5 percent and use the dry residual 95 percent.

6.1.3 Molecular sieves. Solvents *other than methanol* may be dried to a moisture content of 0.05 percent by passing upward through a molecular sieve[11] column, using 1 part molecular sieve per 10 parts of solvent.

7. End Point Detection

7.1 *Color End Point*—The titration to a visual end point is not as accurate or precise as the electrometric end point, and cannot be used for highly colored samples. It may, however, be adequate for routine determinations of water above 0.1 to 0.2 percent, in a relatively colorless system. The end point is taken during titration when the color changes from yellow to orange-red and persists for at least 30 s.

7.2 *Electrometric End Point*—The titration to an electrometric end point is applicable over the entire range of water content, and can be used for samples that give colored solutions. Usually the electrometric end point is determined by the dead-stop technique (3,9).

7.3 There are several commercial titration assemblies on the market, some of which provide a means for automatically stopping the titration at the end point.[12] Necessary operating instructions are provided with these instruments by the manufacturer; therefore, their use is not described here.

7.4 In this method, use of the simple transistorized end point detector is described in 8.2. The complete assembly is described in Appendix A1.

8. Standardization of Karl Fischer Reagent

8.1 Standardize the Karl Fischer reagent daily or as necessary by either the color or electrometric end point, using the amounts of water, sodium tartrate dihydrate, or water-in-methanol shown below:

Water Equivalent *F*, mg/ml	Water, g	Sodium Tartrate Dihydrate, g	Water-in-Methanol, Standard, ml
0.5	0.01 to 0.02	0.1 to 0.15	10 to 20
1	0.03 to 0.05	0.2 to 0.3	30 to 50
3	0.09 to 0.15	0.6 to 0.9	
6	0.18 to 0.30	1.1 to 1.9	

8.2 Add 25 or 50 ml of methanol to a clean, dry titration flask containing a stirring bar. Close the neck of the flask with a two-hole rubber cap. Adjust the magnetic stirrer to give a smooth stirring action. Turn on toggle switch *S2* of the end point detector (see Fig. A2). Set the hi-low toggle switch *S1* to the des red sensitivity. With the electrodes out of the solution or immersed in a wet solution, the meter should read approximately 75 percent of full scale. If the meter reads below 50 percent, the batteries are weak and should be replaced. Titrate with Karl Fischer reagent until a reading of approximately 25 is retained for about 30 s.

8.3 Alternatively, titrate with Karl Fischer reagent to the color end point, 7.1.

8.4 Transfer the selected standard to the pretitrated methanol.

8.4.1 Weigh, to the nearest 0.0001 g, the indicated amount of distilled water from a suitable weighing pipet, hypodermic syringe, or other device, or

8.4.2 Transfer the weighed sodium tartrate dihydrate by means of a dry spatula, dipping the spatula into the alcohol to ensure removal of any adhering tartrate (Note 16), or

8.4.3 Use a hypodermic syringe of suitable capacity to transfer the standard water-in-methanol solution to the titration flask.

NOTE 16—To facilitate transferal of the tartrate to vessels having constricted openings or narrow necks, a spatula with the tip bent at a right angle to the handle is satisfactory. If the tartrate is used for standardizing Karl Fischer reagent for use with samples containing more than 1 percent water, a bias may exist which has been described in Ref. (13)

8.5 Titrate with Karl Fischer reagent to the end point described in 8.2 or 8.3.

8.6 *Calculation*—Calculate the water equivalent, *F*, of the Karl Fischer reagent, in milligrams per milliliter, as follows:

Water as Standard:

$$F = 1000\ G/A$$

Water-in-Methanol as Standard:

$$F = DE/A$$

Sodium Tartrate Dihydrate as Standard:

[11] Linde Type 4A Molecular Sieve has been found satisfactory for this purpose.

[12] Aquameters, Models KF-2 and KF-3, Beckman Instruments, Inc.; Precision-Dow Recordomatic Titrator, Precision Scientific Co.; Metrohm Potentiograph, Brinkmann Instruments, Inc.; Titrator, transistorized, Karl Fischer Magne-Matic, Arthur H. Thomas Co., have been found satisfactory for this purpose.

$$F = (156.6 \times C)/A$$

where:

G = grams of water used,
C = grams of sodium tartrate dihydrate used,
A = milliliters of reagent required for titration of the standard,
D = milliliters of water-in-methanol standard required, and
E = milligrams of water per milliliter in the water-in-methanol standard.

9. Procedure for Soluble Materials, Either Liquid or Solid

9.1 Pipet 25 or 50 ml of the selected sample solvent (Section 5) into the titration flask through the second hole in the rubber cap; see Appendix A1, Section 4.1. Turn on the magnetic stirrer and adjust the speed to give proper stirring. Titrate the water in the solvent with Karl Fischer reagent, as described in 8.2 or 8.3.

9.2 Weigh or pipet a sample of the proper size, usually containing 20 to 300 mg of water, into the flask and titrate again to the same end point.

NOTE 17—The range of water indicated is for macro titrations. Considerably smaller amounts of water can be determined precisely on a micro scale. For example, less than 300 μg of water were titrated in 1-ml samples of benzene by a micro amperometric technique (4).

9.3 *Calculation*—Calculate the water content of the sample as follows:

$$\text{Water, percent} = [(AF \times 0.001)/W] \times 100$$

where:

A = milliliters of reagent require for titration of the sample,
F = water equivalent, in milligrams of water per milliliter of KF reagent, and
W = grams of sample.

10. Procedure for Insoluble Solids

10.1 In case the sample is insoluble in the solvent or solvent mixture used, one of two modifications may be applied. The entire sample - solvent slurry may be titrated, or after stirring and standing, an aliquot of the clear supernatant liquid may be withdrawn and titrated. The latter modification is particularly useful for alkaline samples which are relatively insoluble in the solvent used (12).

10.2 Weigh the sample into a clean and dry titration flask, add 25 to 50 ml of the selected solvent (Section 5) and stopper the flask. Extract the water by stirring with a magnetic stirrer for 15 min or longer, or warming if indicated. Titrate the mixture at room temperature with Karl Fischer reagent as described in 8.2 or 8.3 (Note 18). Also titrate the same volume of solvent as a blank.

NOTE 18—If desired, a known excess of Karl Fischer reagent may be added to the flask, allowed to stand, and then back-titrated with standard water-in-methanol reagent, as described in ASTM Methods D 1348, Test for Moisture in Cellulose.[13] In this case, the electrometric end point indication is the reverse of that described in 8.2.

10.3 *Calculation*—Calculate the water content of the sample as follows:

$$\text{Water, percent} = [((A - B)F \times 0.001)/W] \times 100$$

where:

A = milliliters of reagent required to titrate the sample mixture,
B = milliliters of reagent required to titrate the solvent blank,
F = water equivalent, in milligrams of water per milliliter of KF reagent, and
W = grams of sample.

10.4 Alternatively, add 50 to 100 ml of the solvent to the sample in a volumetric flask, stopper, and extract as before. Make up to the mark, mix, and allow to stand until clear. Transfer a suitable aliquot portion of the supernatant liquid to a titration flask, and titrate with Karl Fischer reagent as described in 8.2 or 8.3. Also titrate the same volume of the solvent, as a blank.

10.5 *Calculation*—Calculate the water content of the sample as follows:

$$\text{Water, percent} = [((A - B)F \times 0.001)/W] \times 100 \times R$$

A = milliliters of reagent required to titrate the sample,
B = milliliters of reagent required to titrate the solvent blank,
F = water equivalent, in milligrams of water per milliliter of KF reagent,
W = grams of sample, and
R = aliquot factor.

11. Precision and Accuracy

11.1 Sensitivity, precision, and accuracy depend on several factors, for example, con-

[13] *Annual Book of ASTM Standards*, Part 21.

centration of the Karl Fischer reagent, titration technique, apparatus, quantity of water titrated, and nature of material being analyzed.

11.2 The sensitivity is about 0.1 mg of water for visual titrations. Less than 0.02 mg can be measured by electrometric titration.

11.3 The following is an example of the precision attained in an interlaboratory (Note 19) study on two samples of acetone containing 0.1 percent and 0.4 percent water and two samples of methyl ethyl ketone containing 0.05 percent and 0.17 percent water:

11.3.1 *Repeatability*—Two results (each the average of duplicate determinations) obtained by the same analyst should be considered suspect if they differ by more than 0.013 percent, absolute (95 percent confidence level). Duplicate determinations which agree within 0.008 percent are acceptable for averaging (95 percent confidence level).

11.3.2 *Reproducibility*—Two results (each the average of duplicate determinations) obtained by analysts in different laboratories should be considered suspect if they differ by more than 0.028 percent, absolute (95 percent confidence level).

NOTE 19—The interlaboratory study was carried out by ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products, Subcommittee V on Solvents, Plasticizers, and Chemical Intermediates. Seven laboratories participated, with a single analyst performing duplicate determinations on each of two days, using two methods on the four samples described above. ASTM Method D 1364 Test for Water in Volatile Solvents (Fischer Reagent Titration Method)[8] was the subject of the test program being compared with each laboratory's own version of a Karl Fischer method. As neither the means nor the variances of the two sets of data proved significantly different, all of the results were pooled to give estimates of the repeatability based on 55 degrees of freedom and reproducibility based on 47 degrees of freedom.

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APPENDIXES

A1. SUGGESTED APPARATUS FOR KARL FISCHER METHOD

A1.1 Scope

A1.1.1 Described in this Appendix is a glass apparatus, its assembly, and design of a dual-transistorized end point detector. There are available a number of assemblies of similar design which are equally suitable for storing and dispensing the Karl Fischer reagent, for containing the sample, and for detecting the end point. See Appendix A3 and References (1,6).

A1.2 Titration Assembly

A1.2.1 The storage and dispensing assembly

shall consist of the following parts (see Fig. A1):

A1.2.1.1 *Buret*, automatic, with TFE-fluorocarbon resin plug and automatic zero, reservoir bottle, and connecting tube.[14] Select the size buret and bottle needed. An overhead reservoir[15] with micro buret may also be used.

A1.2.1.2 *Tube, Drying*, calcium chloride, one bulb, 200 mm long.

A1.2.1.3 *Bottle, Aspirator*, with outlet for tubing connections, 500-ml capacity.

A1.2.1.4 *Stirrer, Magnetic*, with stirring bar coated with TFE-fluorocarbon resin.

A1.2.1.5 *Flask, Titration*, 250-ml capacity.[16]

A1.2.1.6 *Electrodes*, with connecting cord and plugs.[17]

A1.2.1.7 *Rubber Cap*, 1.5 in. (38 mm) in outside diameter.[18] Punch two holes, 3 to 4 mm in diameter, through the cap.

A1.3 End Point Detector

A1.3.1 Figure A2 shows the wiring diagram of a sensitive end point detector and a parts list. This end point detector is a dual-range constant-current type detector. The electrode current is switched to select either 5 μA or 100 μA. This makes possible Karl Fischer titrations using a weak reagent for low ppm water determinations or the usual titrations using the stronger reagents.

A1.4 Assembly of Apparatus

A1.4.1 Assemble the apparatus as shown in Fig. A1. Fill the drying tube and aspirator bottle with dessicant.[19] Insert the buret tip through one hole in the rubber cap. Use the other hole for inserting a pipet or hypodermic syringe containing liquid samples. Under humid conditions, keep the second hole plugged except when introducing a sample, or pass a slow stream of dry nitrogen into the flask. Connect the plugs on ends of the leads on the titration flask to the jacks of the end point detector.

A2. INTERFERING COMPOUNDS THAT REACT STOICHIOMETRICALLY WITH KF REAGENT THEREBY ENABLING FREE WATER TO BE CALCULATED AFTER APPLYING CORRECTION

A2.1 Many interfering substances react stoichiometrically with constituents of the KF reagent. Consequently, when independent analyses can be made for these compounds, suitable corrections can be applied to the apparent water results. Also in many cases moisture can be separated from the interfering substance by extraction with a water-miscible liquid in which the sample is insoluble or by distillation, preferably using a carrier that forms a homogeneous azeotrope, for example, dioxane, ethanol - benzene. Materials in this class are given in Table A1.

A2.2 Some compounds react only partially with KF reagent when titrated under normal conditions. These include the following:

Methylolurea[20]	Dichromates
Peroxides, diacyl[21]	Iron oxide
Peracids[21]	Nickel oxide
Quinone	Sodium peroxide
Arsenious oxide	Sodium sulfide
Chromates	

A3. OTHER ASTM KARL FISCHER REAGENT WATER METHODS

Designation	Sponsoring Committee	Title of Method
D 789	D-20	Specification for Nylon Injection Molding and Extrusion Materials[22]
D 890	D-17	Test for Water in Liquid Naval Stores[9]
D 1123	D-15	Test for Water in Engine Coolant Concentrate by the Iodine Reagent Method[23]
D 1348	D-23	Tests for Moisture in Cellulose[24]
D 1364	D-1	Test for Water in Volatile Solvents (Fischer Reagent Titration Method)[9]
D 1457	D-20	Specification for TFE-Fluorocarbon Resin Molding and Extrusion Materials[22]
D 1533	D-9	Test for Water in Insulating Liquids (Karl Fischer Method)[24]
D 1568	D-12	Sampling and Chemical Analysis of Alkylbenzene Sulfonates[25]
D 1631	D-16	Test for Water in Phenol and Related Materials by the Iodine Reagent Method[9]
D 1638	D-20	Testing Urethane Foam Isocyanate Raw Materials[9]
D 1744	D-2	Test for Water in Liquid Petroleum Products by Karl Fischer Reagent[10]
D 2072	D-1	Test for Water in Fatty Nitrogen Compounds[9]

[14] Kimble Glass Co. Catalog No. 17124F, has been found satisfactory for this purpose.

[15] Kimble Glass Co., Catalog No. 17138F, has been found satisfactory for this purpose.

[16] A. H. Thomas Co. Catalog No. 9682-K-40 titration cell has been found satisfactory for this purpose.

[17] A. H. Thomas Co. Catalog No. 9682-K70 electrode has been found satisfactory for this purpose.

[18] Devol Rubber Co. Catalog No. 270 or E. H. Sargent Co. Catalog No. S-73185, 1.5 in., have been found satisfactory for this purpose.

[19] Indicating-type Drierite has been found satisfactory for this purpose.

[20] Interference of methylolurea can be eliminated by titration at −40 C (1).

[21] Diacyl peroxides and peracids fairly rapidly oxidize the HI of spent KF reagent. After a short time interval following addition of KF reagent, this reaction may be quantitative (1).

[22] *Annual Book of ASTM Standards*, Part 36.

[23] *Annual Book of ASTM Standards*, Part 30.

[24] *Annual Book of ASTM Standards*, Part 40.

A4. DETERMINATION OF WATER IN GASES

A4.1 Procedures for determining moisture in gases are described in the literature (1,5,6,10,11).

A4.2 As mentioned in Section 1, this method does not include procedures for samples that are gaseous at room temperature. The safe handling and analysis of gases requires a thorough knowledge of their properties and also the use of special apparatus and techniques. The moisture content may range from 1000 down to 2 to 3 ppm.

A4.3 The manufacturers of gases have developed very precise Karl Fischer procedures for measuring moisture down to a few parts per million (10,11). They should be consulted when need arises. Also, there are available commercial instruments that operate on the dew point, infrared, conductance, electrolysis principle, etc., which are rapid and accurate for determining moisture in gas samples (1,6).

TABLE A1 Materials Reacting Stoichiometrically With KF Reagent

Class or Compound	Moles of Apparent H_2O per Mole Compound	Do Not React
Ascorbic acid	1	
Hydrazine derivatives	1	Hydrazine sulfate
Mercaptans	0.5	
Silanols, R_3SiOH	1	
$R_2Si(OH)_2$	2	
Arsenate, $RAsO_4$	3	
Arsenite, $RAsO_3$	2	
Boric acid, H_3BO_3	3	
HBO_2	2	
Boric oxide, B_2O_3	3	
Bicarbonates	1	
Carbonates	1	Calcium carbonate
Cupric salts	0.5	
Ferric salts	0.5	Ferric fluoride
Metal hydroxides, MOH	1	
$M(OH)_2$	2	
Metal oxides, CaO, MgO, ZnO, Ag_2O, HgO, Cu_2O, MnO_2, PbO[a], PbO_2[a]	1	Aluminum, cupric, barium oxides
Pb_3O_4	3	
Pyrosulfites	1	Pyrosulfate
Sodium nitrite[b]	0.5	
Sulfites	1	Hyposulfite
Stannous salts	1	
Tetraborate	7	
Thiosulfate	0.5	

[a] The lead oxides react only partially when dispersed in methanol, probably because of insolubility. In acetic acid solution, however, these oxides react quantitatively.

[b] Reaction is fairly slow. Apparently free water can be determined in presence of $NaNO_2$ by rapid titration with KF reagent.

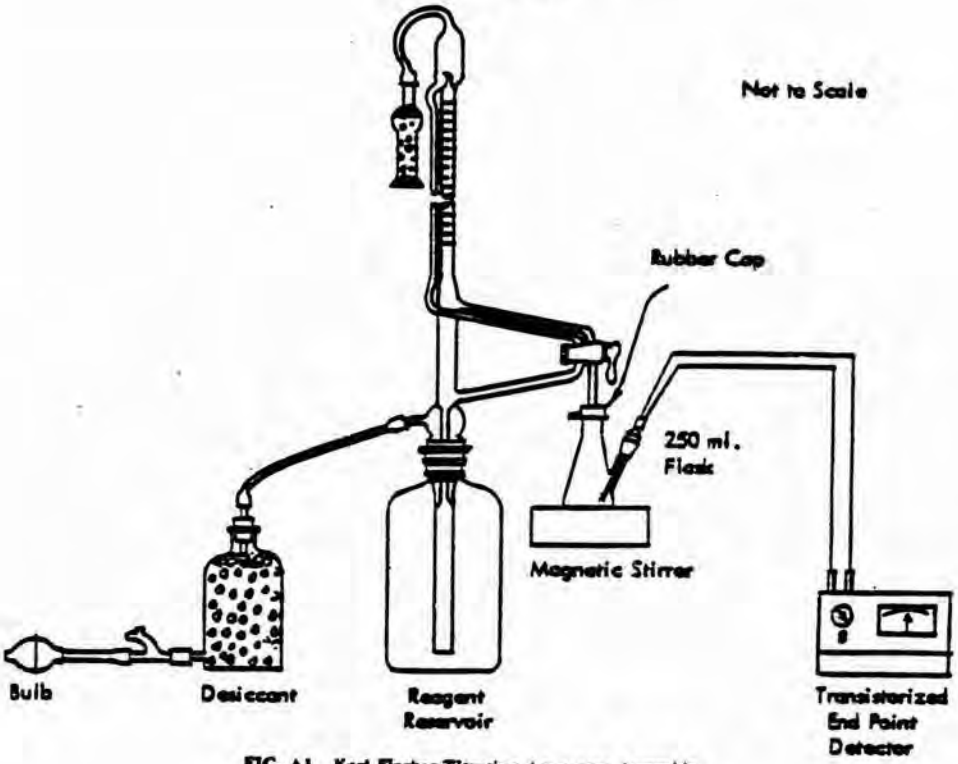
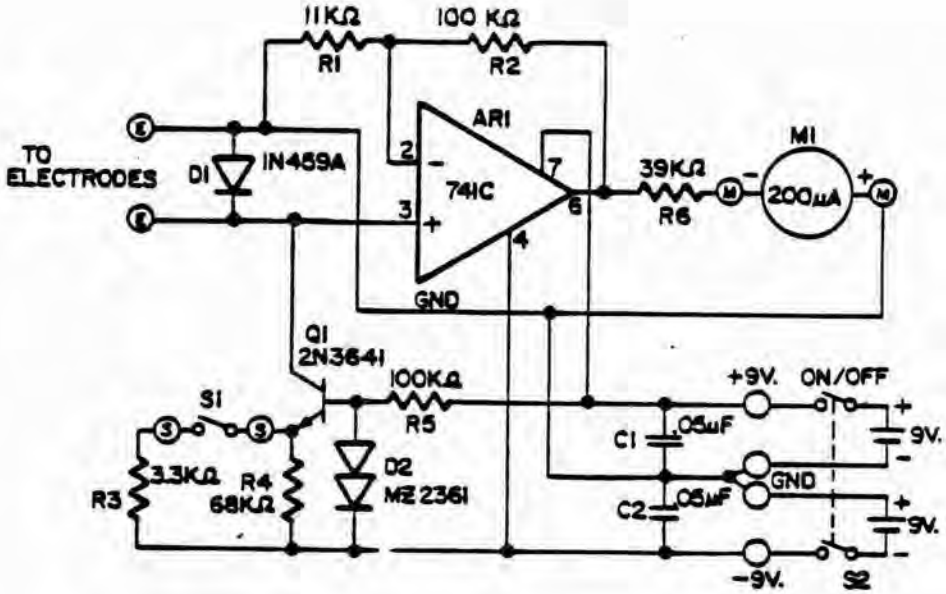


FIG. A1 Karl Fischer Titration Apparatus Assembly.



PARTS LIST

METER SIMPSON MODEL 524
(4½ in.; 0–200 μA)
Amplifier 741C
Diode 1N 459A
Diode MZ 2361
Transistor 2N 3641
Capacitor C1, 0.05 μF
Capacitor C2, 0.05 μF
Resistor R1, 11 KΩ, ¼ W carbon
Resistor R2, 100KΩ, ¼ W carbon
Resistor R3, 3.3 KΩ, ¼ W carbon
Resistor R4, 68 KΩ, ¼ W carbon
Resistor R5, 100 KΩ, ¼ W carbon
Resistor R6, 39 KΩ, ¼ W carbon
Switch *S1*, SPST Toggle
Switch *S2*, DPST Toggle
2 Battery 9 V.
2 Battery holders
Chassis, aluminum
Connector, Amphenol
Printed circuit board

NOTE—All resistors ¼-W carbon *S1* open for 5 μA. Closed for 100 μA.

FIG. A2. Circuit Diagram of Sensitive End Point Detector.

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APPENDIX C.2

ADDITIONAL WASTE TESTING PARAMETERS

Analytical Methods[1]

100 PHYSICAL PROPERTIES

Color
- Colorimetric, ADMI.....Method 110.1
- Colorimetric, Platinum-Cobalt.....Method 110.2
- Spectrophotometric.....Method 110.3

Conductance
- Specific Conductance.....Method 120.1

Hardness, Total (mg/l as $CaCO_3$)
- Colorimetric, Automated EDTA.....Method 130.1
- Titrimetric, EDTA.....Method 130.2

Odor
- Threshold Odor (Consistent Series).....Method 140.1

pH
- Electrometric.....Method 150.1

Residue
- Filterable
  - Gravimetric, Dried at 180°C.....Method 160.1
- Non-Filterable
  - Gravimetric, Dried at 103-105°C.....Method 160.2
- Total
  - Gravimetric, Dried at 103-105°C.....Method 160.3
- Volatile
  - Gravimetric, Ignition at 550° .....Method 160.4
- Settleable Matter
  - Volumetric, Imhoff Cone.....Method 160.5

Temperature
- Thermometric.....Method 170.1

Turbidity
- Nephelometric.....Method 180.1

200 METALS

Atomic Absorption Methods.....Method 200.0

Aluminum
- AA, Direct Aspiration.....Method 202.1
- AA, Furnace.....Method 202.2

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[1] U.S. EPA, "Methods for Chemical Analysis of Water and Wastes," Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1979.

200 METALS (Cont.)

Antimony
AA, Direct Aspiration.....Method 204.1
AA, Furnace.....Method 204.2

Arsenic
AA, Furnace.....Method 206.2
AA, Hydride.....Mehtod 206.3
Spectrophotometric, SDDC.....Method 206.4
Digestion Method for Hydride and SDDC.....Method 206.5

Barium
AA, Direct Aspiration.....Method 208.1
AA, Furnace.....Method 208.2

Beryllium
AA, Direct Aspiration.....Method 210.1
AA, Furnace.....Method 210.2

Boron
Colorimetric, Curcumin.....Method 212.3

Cadmium
AA, Direct Aspiration.....Method 213.1
AA, Furnace.....Method 213.2

Calcium
AA, Direct Aspiration.....Method 215.1
Titrimetric, EDTA.....Method 215.2

Chromium
AA, Direct Aspiration.....Method 218.1
AA, Furnace.....Method 218.2
Chelation-Extraction.....Method 218.3
Hexavalent, Chelation-Extraction.....Method 218.4

Cobalt
AA, Direct Aspiration.....Method 219.1
AA, Furnace.....Method 219.2

Copper
AA, Direct Aspiration.....Method 220.1
AA, Furnace.....Method 220.2

200 METALS (Cont.)

Gold
AA, Direct Aspiration.....Method 231.1
AA, Furnace.....Method 231.2

Iridium
AA, Direct Aspiration.....Method 235.1
AA, Furnace.....Method 235.2

Iron
AA, Direct Aspiration.....Method 236.1
AA, Furnace.....Method 236.2

Lead
AA, Direct Aspiration.....Method 239.1
AA, Furnace.....Method 239.2

Magnesium
AA, Direct Aspiration.....Method 242.1

Manganese
AA, Direct Aspiration.....Method 243.1
AA, Furnace.....Method 243.2

Mercury
Cold Vapor, Manual.....Method 245.1
Cold Vapor, Automated.....Method 245.2
Cold Vapor, Sediments.....Method 245.5

Molybdenum
AA, Direct Aspiration.....Method 246.1
AA, Furnace.....Method 246.2

Nickel
AA, Direct Aspiration.....Method 249.1
AA, Furnace.....Method 249.2

Osmium
AA, Direct Aspiration.....Method 252.1
AA, Furnace.....Method 252.2

Palladium
AA, Direct Aspiration.....Method 253.1
AA, Furnace.....Method 253.2

200 METALS (Cont.)

Platinum
AA, Direct Aspiration.....Method 255.1
AA, Furnace.....Method 255.2

Potassium
AA, Direct Aspiration.....Method 258.1

Rhenium
AA, Direct Aspiration.....Method 264.1
AA, Furnace.....Method 264.2

Rhodium
AA, Direct Aspiration.....Method 265.1
AA, Furnace.....Method 265.2

Ruthenium
AA, Direct Aspiration.....Method 267.1
AA, Furnace.....Method 267.2

Selenium
AA, Furnace.....Method 270.2
AA, Hydride.....Method 270.3

Silver
AA, Direct Aspiration.....Method 272.1
AA, Furnace.....Method 272.2

Sodium
AA, Direct Aspiration.....Method 273.1

Thallium
AA, Direct Aspiration.....Method 279.1
AA, Furnace.....Method 279.2

Tin
AA, Direct Aspiration.....Method 282.1
AA, Furnace.....Method 282.2

Titanium
AA, Direct Aspiration.....Method 283.1
AA, Furnace.....Method 283.2

Vanadium
AA, Direct Aspiration.....Method 286.1
AA, Furnace.....Method 286.2

200 METALS (Cont.)

Zinc
- AA, Direct Aspiration.....Method 289.1
- AA,Furnace.....Method 289.2

300 INORGANIC, NON-METALS

Acidity
- Titrimetric.....Method 305.1

Alkalinity
- Titrimetric (pH 4.5).....Method 310.1
- Colorimetric, Automated Methyl Orange.....Method 310.2

Bromide
- Titrimetric.....Method 320.1

Chloride
- Colorimetric, Automated Ferricyanide, AA I.....Method 325.1
- Colorimetric, Automated Ferricyanide, AA II.....Method 325.2
- Titrimetric, Mercuric Nitrate.....Method 325.3

Chlorine, Total Residual
- Titrimetric, Amperometric.....Method 330.1
- Titrimetric, Back-Iodometric.....Method 330.2
- Titrimetric, Iodometric.....Method 330.3
- Titrimetric, DPD-FAS.....Method 330.4
- Spectrophotometric, DPD.....Method 330.5

Cyanide
- Amenable to Chlorination
  - Titrimetric, Spectrophotometric.....Method 335.1
- Total
  - Titrimetric, Spectrophotometric.....Method 335.2
  - Colorimetric, Automated UV.....Method 335.3

Fluoride
- Colorimetric, SPADNS with Bellack Distillation.....Method 340.1
- Potentiometric, Ion Selective Electrode.....Method 340.2
- Colorimetric, Automated Complexone.....Method 340.3

Iodide
- Titrimetric.....Method 345.1

Nitrogen (Cont.)
- Kjeldahl, Total
  - Colorimetric, Automated Phenate.....Method 351.1
  - Colorimetric, Semi-Automated Block Digester AAII.....Method 351.2
  - Colorimetric; Titrimetric; Potentiometric.....Method 351.3
  - Potentiometric, Ion Selective Electrode.....Method 351.4

Nitrate
- Colorimetric, Brucine.....Method 352.1

Nitrate-Nitrite
- Colorimetric, Automated Hydrazine Reduction.....Method 353.1
- Colorimetric, Automated Cadmium Reduction.....Method 353.2
- Colorimetric, Manual Cadmium Reduction.....Method 353.3

Nitrite
- Spectrophotometric.....Method 354.1

Oxygen, Dissolved
- Membrane Electrode.....Method 360.1
- Modified Winkler (Full Bottle Technique).....Method 360.2

Phosphorus
- All Forms
  - Colorimetric, Automated, Ascorbic Acid.....Method 365.1
  - Colorimetric, Ascorbic Acid, Single Reagent.....Method 365.2
  - Colorimetric, Ascorbic Acid, Two Reagent.....Method 365.3
- Total
  - Colorimetric, Automated, Block Digestor,AA II.....Method 365.4

Silica, Dissolved
- Colorimetric.....Method 370.1

Sulfate
- Colorimetric, Automated Chloranilate.....Method 375.1
- Colorimetric, Automated Methyl Thymol Blue, AA II.....Method 375.2
- Gravimetric.....Method 375.3
- Turbidimetric.....Method 375.4

Sulfide
- Titrimetric, Iodine.....Method 376.1
- Colorimetric, Methylene Blue.....Method 376.2

300 INORGANIC, NON METALLICS (Cont.)

Sulfite
- Titrimetric.....Method 377.1

400 ORGANICS

Biochemical Oxygen Demand
- BOD (5 day, 20°C).....Method 405.1

Chemical Oxygen Demand
- Titrimetric, Mid-Level.....Method 410.1
- Titrimetric, Low Level.....Method 410.2
- Titrimetric, High Level for Saline Waters.....Method 410.3
- Colorimetric, Automated; Manual.....Method 410.4

Oil and Grease, Total Recoverable
- Gravimetric, Separatory Funnel Extraction.....Method 413.1
- Spectrophotometric, Infrared.....Method 413.2

Organic Carbon, Total
- Combustion or Oxidation.....Method 415.1

Petroleum Hydrocarbons, Total, Recoverable
- Spectrophotometric, Infrared.....Method 418.1

Phenolics, Total Recoverable
- Spectrophotometric, Manual 4-AAP with Distillation.....Method 420.1
- Colorimetric, Automated 4-AAP with Distillation.....Method 420.2
- Spectrophotometric, MBTH with Distillation.....Method 420.3

Methylene Blue Active Substances (MBAS)
- Colorimetric.....Method 425.1

NTA
- Colorimetric, Manual, Zinc-Zincon.....Method 430.1
- Colorimetric, Automated, Zinc-Zincon,.....Method 430.2

Note: Other organic chemicals will be analyzed according to the appropriate gas chromatograph (GC) analytical procedure described in "Test Methods for Evaluating Solid Waste; Physical/Chemical Methods," (U.S. EPA, 1980).

APPENDIX C.3

SAMPLING EQUIPMENT AND PROCEDURES

SAMPLING EQUIPMENT

Introduction

Sampling the diverse types of RCRA regulated wastes requires a variety of different types of samplers. A number of such sampling devices are described in this section. While some of these samplers are commercially available, others will have to be fabricated by the user. Table 3.2-1 is a general guide to the types of waste that can be sampled by each of the samplers described.

Table 3.2-1

SAMPLING EQUIPMENT FOR PARTICULAR WASTE TYPES

Sampling Point / Waste Type	Drum	Sacks and Bags	Open Bed Truck	Closed Bed Truck	Storage Tanks or Bins	Waste Piles	Ponds, Lagoons & Pits	Con-veyor Belt	Pipe
Free Flowing Liquids and Slurries	Coli-wasa	N/A	N/A	Coli-wasa	Weighted Bottle	N/A	Dipper	N/A	Dip-per
Sludges	Trier	N/A	Trier	Trier	Trier	N/A		N/A	N/A
Moist Powders or Granules	Trier	Trier	Trier	Trier	Trier	Trier	Trier	Shovel	
Dry Powders or Granules	Thief	Thief	Thief	Thief	Thief	Thief	Thief	Shovel	
Sand or Packed Powders and Granules	Auger	Auger	Auger	Auger				N/A	
Large Grained Solids	arge Trier	Large Trier	Large Trier	Large Trier	Large Trier	Large Trier	Large Trier	Large Trier	

3.2.1 Composite Liquid Waste Sampler (Coliwasa)

Scope and Purpose

The Coliwasa is a device employed to sample free flowing liquids and slurries contained in drums, shallow open top tanks, pits and similar containers. It is especially useful in sampling wastes which consist of a number of immiscible liquid phases.

The Coliwasa consists of a glass, plastic, or metal tube equipped with an end closure which can be opened and closed while the tube is submerged in the material to be sampled.

The Coliwasa was developed by the California Department of Health under a grant from the U.S. EPA and their report, "Samplers and Sampling Procedures for Hazardous Waste Streams" [Appendix I of this manual] should be consulted.

General Comments and Precautions

1. Do not use a plastic Coliswasa to sample wastes containing organic materials.
2. Do not use a glass Coliwasa to sample liquids that contain hydrofluoric acid.
3. If significant amounts of solid material are present within 2 inches of the bottom of the container to be sampled, special procedures will be necessary to obtain a representative sample of this solid phase.

Apparatus

Coliwasas are not available commercially and must be fabricated to conform to the specifications detailed in Figure 3.2-1. Table 3.2-2 lists the parts required to fabricate a plastic or glass Coliwasa.

Table 3.2-2

PARTS FOR CONSTRUCTING A COLIWASA

Quantity	Item	Comments	Supplier
1	Sample tube, translucent PVC plastic, 4.13 cm I.D. x 1.52 m long x 0.4 cm wall thickness	Plastic Coliwasa only	Plastic supply houses
1	Sample tube, borosilicate glass, 4.13 cm I.D. x 1.52 m long	Glass Coliwasa only	Corning Glass Works #72-1602
1	Stopper, neoprene rubber #9		Laboratory supply house
1	Stopper rod, PVC, 0.95 cm O.D. x 1.67 m long	Plastic Coliwasa only	Plastic supply houses
1	Stopper rod, teflon, 0.95 cm O.D. x 1.67 m long	Glass or Plastic Coliwasa	Plastic supply houses
1	Locking block, PVC, 3.8 cm O.D. x 10.2 cm long with 0.56 cm hole in center	Fabricate by drilling 0.56 cm hole through center	Plastic supply houses
1	Locking block sleeve, PVC 4.13 cm I.D. x 6.35 cm long	Fabricate from stock 4.13 cm PVC pipe	Plastic supply houses
1	T-handle, aluminum, 18 cm long x 2.86 cm wide with 1.27 cm wide channnel	Fabricate from aluminum bar stock	Hardware stores

Quantity	Item	Comments	Supplier
1	Swivel, aluminum bar 1.27 cm square x 5.08 cm long with 3/8" NC inside thread to attach stopper rod	Fabricate from aluminum bar stock	hardware stores
1	Nut, PVC, 3/8" NC		Plastic supplier
1	Washer, PVC, 3/8" NC		Plastic supplier
1	Nut, stainless steel, 3/8" NC		Hardware stores
1	Washer, stainless steel, 3/8"		Hardware stores
1	Bolt, 3.12 cm long x 3/16" NC		Hardware stores
1	Nut, 3/16" NC		Hardware stores
1	Washer, lock 3/16"		Hardware stores

Assembly

Assemble sampler as follows:

1. Attach swivel to the T-handle with the 3.12cm long bolt and secure with the 3/16" NC washer and lock nut.
2. Shape stopper into a cone by boring a 0.95 cm hole through the center of the stopper. Insert a short piece of 0.95 cm O.D. handle through the hole until the end of the handle is flush against the bottom (smaller diameter) surface of the stopper. Carefully and uniformly turn the stopper into a cone against a grinding wheel. This is done by turning the stopper with the handle and grinding it down conically from about 0.5 cm of the top (larger diameter) surface to the edge of the 0.95 cm hole on the bottom surface. Attach neoprene stopper to one end of the stopper rod and secure with the 3/8" NC washer and lock nut.
3. Install the stopper and stopper rod assembly in the sampling tube.
4. Secure locking block sleeve on the block with glue or screws.
5. Position the locking block on top of the sampling tube so that the sleeveless portion of the block fits inside the tube, the sleeve sits against the top end of the tube, and the upper end of the stopper rod slips through the center hole of the block.
6. Attach the upper end of the stopper to the swivel of the T-handle.

7. Place the sampler in the closed position and adjust the tension on the stopper by screwing the T-handle in or out.
8. Test the tension by filling the Coliwasa with water to insure it is leak free.

Procedure

1. Clean Coliwasa.
2. Adjust sampler's locking mechanism to insure that the stopper provides a tight closure. Open sampler by placing stopper rod handle in the T-position and pushing the rod down until the handle sits against the sampler's locking block.
3. Slowly lower the sampler into the waste at a rate which permits the level of liquid inside and outside the sampler to remain the same. If the level of waste in the sampler tube is lower inside than outside, the sampling rate is too fast and will produce a non-representative sample.
4. When the sampler hits the bottom of the waste container, push sampler tube down to close and lock the stopper by turning the T-handle until it is upright and one end rests on the locking block.
5. Withdraw Coliwasa from waste and wipe the outside with a disposable cloth or rag.
6. Place sample tube at mouth of a container and discharge sample by slowly opening the sampler.

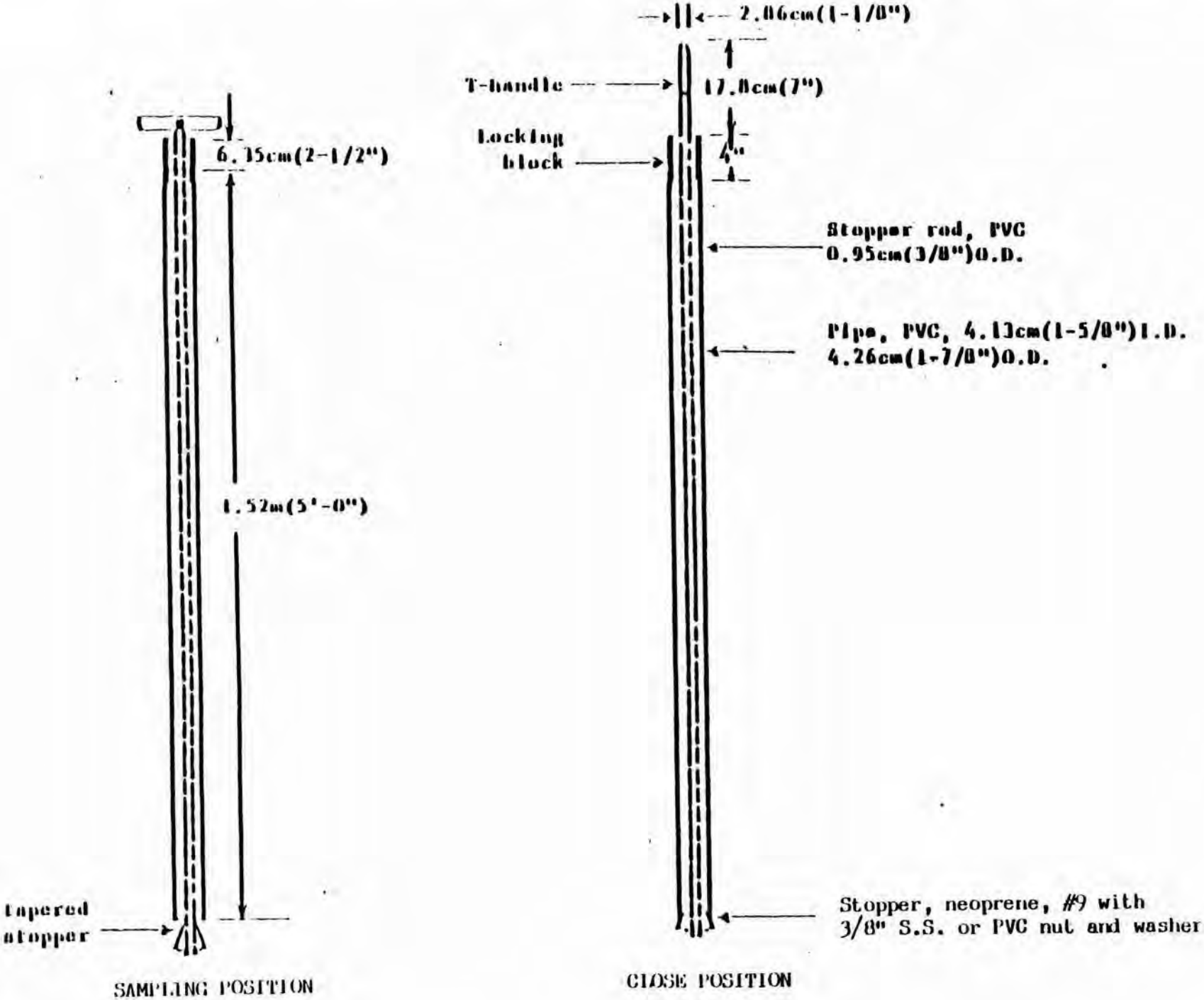

2.86cm(1-1/8")
T-handle
17.8cm(7")
Locking block
4"
6.35cm(2-1/2")
Stopper rod, PVC
0.95cm(3/8")O.D.
Pipe, PVC, 4.13cm(1-5/8")I.D.
4.26cm(1-7/8")O.D.
1.52m(5'-0")
tapered stopper
Stopper, neoprene, #9 with
3/8" S.S. or PVC nut and washer
SAMPLING POSITION
CLOSE POSITION

Figure 3.2-1

3.2.2 Weighted Bottle

Scope and Application

The sampler consists of a glass or plastic bottle, sinker, stopper and a line which is used to lower and raise the bottle and to open the bottle. A weighted bottle samples liquids and free flowing slurries.

General Comments and Precautions

1. Do not use a plastic bottle to sample wastes containing organic materials.
2. Do not use a glass bottle to sample wastes that contain hydrofluoric acid.
3. Before sampling insure that the waste will not corrode the sinker, bottle holder or line.

Apparatus

A weighted bottle with line is built to the specifications in ASTM Methods D 270 and E 300 (Figure 3-2-2).

Procedure

1. Clean bottle.
2. Assemble weighted bottle sampler.
3. Lower the sampler to directed depth and pull out the bottle stopper by jerking the line.
4. Allow bottle to fill completely as evidenced by cessation of air bubbles.
5. Raise sampler, cap and wipe off with a disposable cloth. The bottle can serve as a sample container.

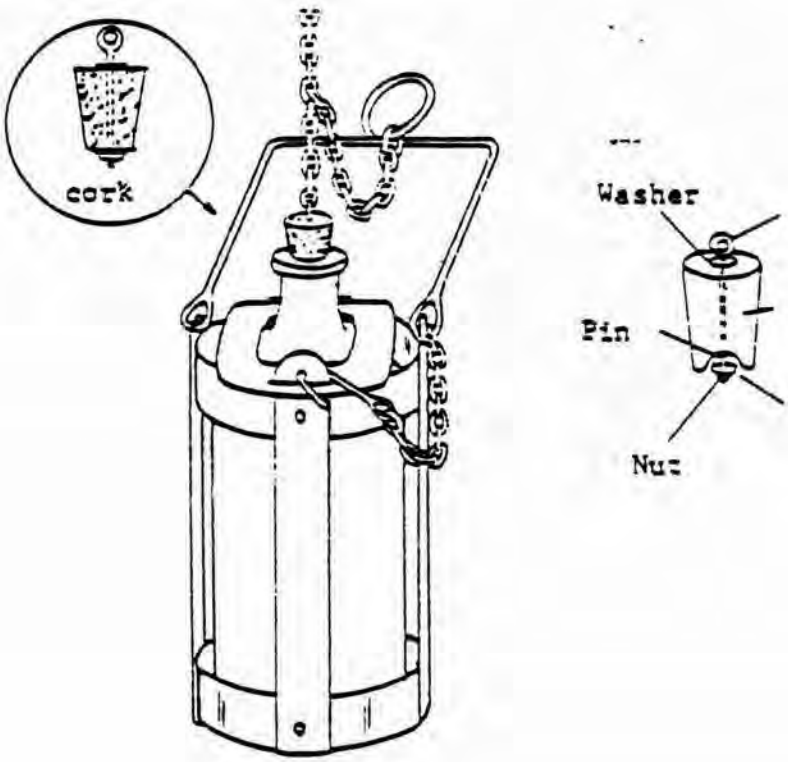


Figure 3.2
WEIGHTED BOTTLE SAMPLER

3.2.3 Dipper

Scope and Application

The dipper consists of a glass or plastic beaker clamped to the end of a 2 or 3 piece telescoping aluminum or fiberglass pole which serves as the handle. A dipper samples liquids and free flowing slurries.

General Comments and Precautions

1. Do not use a plastic beaker to sample wastes containing organic materials.
2. Do not use a glass beaker to sample wastes of high pH or which contain hydrofluoric acid.
3. Paint aluminum pole and clamp with a 2 part epoxy or other chemical resistant paint when sampling either alkaline or acidic wastes.

Apparatus

Dippers are not available commercially and must be fabricated to confrom to the specifications detailed in Figure 3.2-3. Table 3.2-3 lists the parts required to fabricate a dipper.

Table 2.2-3
PARTS FOR CONSTRUCTING A DIPPER

Quantity	Item	Supplier
1	Adjustable clamp, 6.4 to 8.9 cm (2 1/2 to 3 1/2") for 250 to 600 ml beakers. Heavy duty aluminum.	Laboratory supply houses
1	Tube 2.5 to 4.5 meters long with joint cam locking mechanism. Diameter 2.54 cm ID and 3.18 cm ID.	Swimming pool supply houses

Quantity	Item	Supplier
1	Polypropylene or glass beaker, 250 ml to 600 ml.	Laboratory supply houses
4	Bolts 2 1/4" x 1/4", NC	Hardware stores
4	Nuts, 1/4", NC	Hardware stores

Procedure

1. Clean beaker, clamp, and handle.
2. Assemble dipper by bolting adjustable clamp to the pole. Place beaker in clamp and fasten shut.
3. Turn dipper so the mouth of the beaker faces down and insert into waste material. Turn beaker right side up when dipper is at desired depth. Allow beaker to fill completely as shown by the cessation of air bubbles.
4. Raise dipper and transfer sample to container.

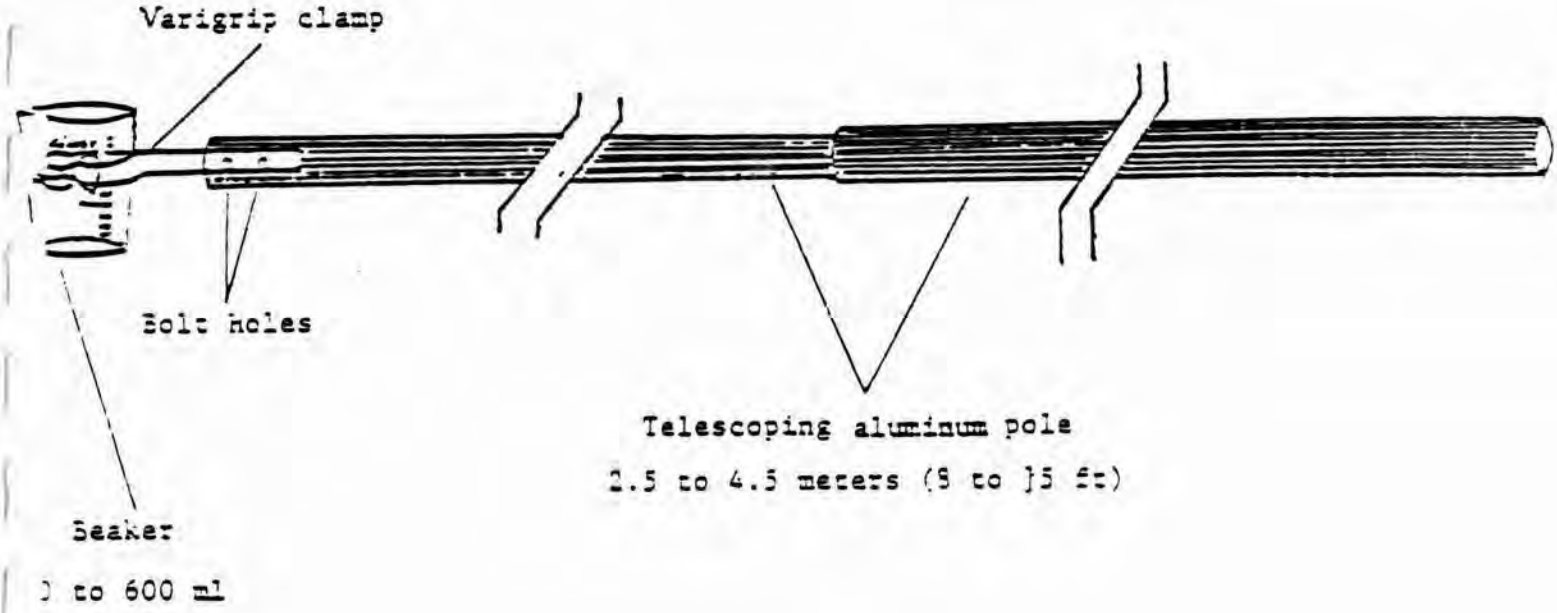


Figure 3.2-3
DIPPER

3.2.4 Thief

Scope and Application

A thief consists of two slotted concentric tubes usually made of stainless steel or brass. The outer tube has a conical pointed tip which permits the sampler to penetrate the material being sampled. The inner tube is rotated to open and close the sampler. A thief is used to sample dry granules or powdered wastes whose particle diameter is less than 1/3 the width of the slots.

Apparatus

A thief is available at laboratory supply stores. (Figure 3.2-4)

Procedure

1. Clean sampler.
2. Insert closed thief into waste material. Rotate inner tube to open thief. Wiggle the unit to encourage material to flow into thief. Close thief and withdraw. Place sampler thief in a horizontal position with the slots facing upward. Remove inner tube from thief and transfer sample to a container.

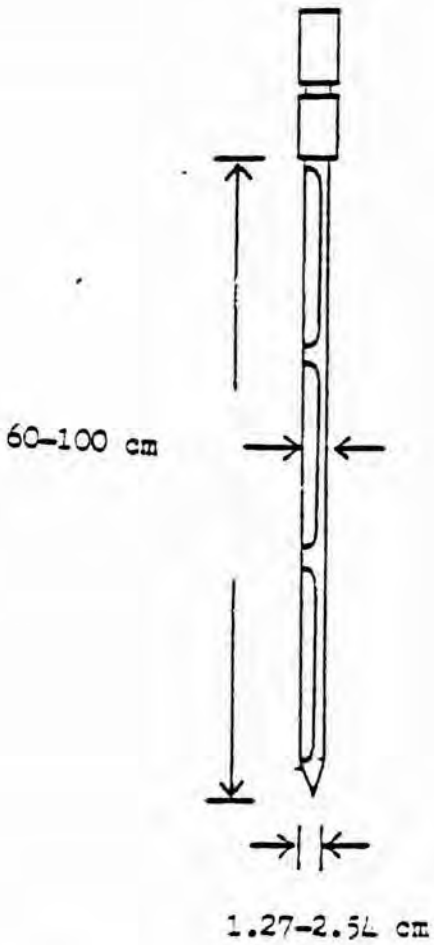


Figure 3.2-4
THIEF SAMPLER

3.2.5 Trier

Scope and Application

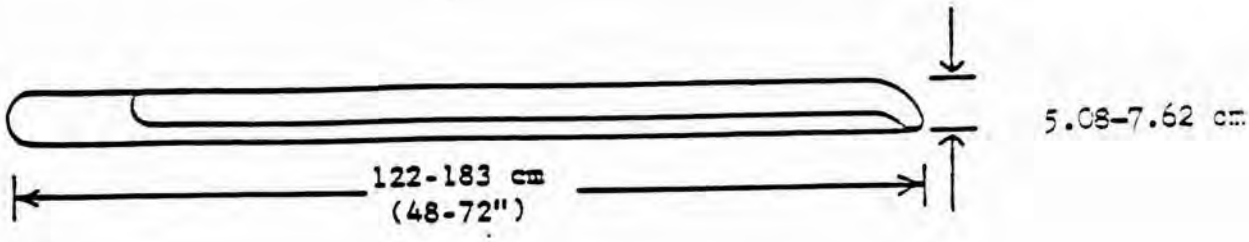
A trier consists of a tube cut in half lengthwise with a sharpened tip that allows the sampler to cut into sticky solids and loosen soil. A trier samples moist or sticky solids with a particle diameter less than 1/2 the diameter of the trier.

Apparatus

1. Triers 61 to 100 cm long and 1.27 to 2.54 cm in diameter are available at laboratory supply stores.
2. A large trier can be fabricated to confrom to the specifications in Figure 3-5. A metal or polyvinyl chloride pipe 1.52 m (5') long x 3.2 cm (1.4") I.D. with a 0.32 cm (1 1/8") wall thickness is needed. The pipe should be sawed lengthwise, about 60-40 split, to form a trough stretching from one end to 10 cm away from the other end. The edges of the slot and the tip of the pipe are sharpened to permit the sampler to cut into the waste material being sampled. The unsplit length of the pipe serves as the handle.

Procedure

1. Clean trier.
2. Insert trier into waste material 0 to 45° from horizontal. Rotate trier to cut a core of the waste. Remove trier with concave side up and transfer sample to container.



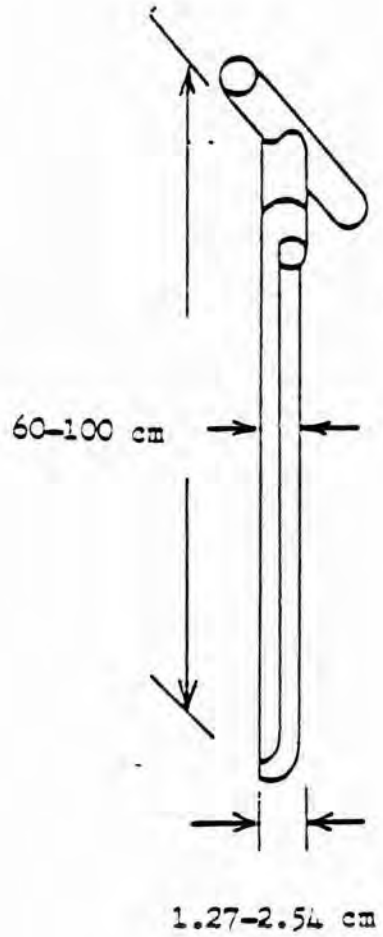


Figure 3.2-5
SAMPLING TRIERS

3.2.6 Auger

Scope and Application

An auger consists of sharpened spiral blades attached to a hard metal central shaft. An auger samples hard or packed solid wastes or soil.

Apparatus

Augers are available at hardware and laboratory supply stores.

Procedures

1. Clean sampler.
2. Bore a hole through the middle of an aluminum pie pan large enough to allow the blade of the auger to pass through. The pan will be used to catch the sample brought to the surface by the auger.
3. Place pan against the sampling point. Auger through the hole in the pan until the desired sampling depth is reached. Back off the auger and transfer the sample in the pan and adhering to the auger to a container. Spoon out the rest of the loosened sample with a sample trier.

3.2.7 Scoop and Shovel

Scope and Application

Scoops and shovels are used to sample granular or powdered material in bins, shallow containers and conveyor belts.

Apparatus

Scoops are available at laboratory supply houses. Flat nosed shovels are available at hardware stores.

Procedure

1. Clean sampler.
2. Obtain a full cross section of the waste material with the scoop or shovel large enough to contain the waste collected in one cross section sweep.

Sub-Section 3.3

SAMPLE CONTAINERS

Containers

The most important factors to consider when choosing containers for hazardous waste samples are compatibility with the waste, cost, resistance to breakage, and volume. Containers must not distort, rupture, or leak as a result of chemical reactions with constituents of waste samples. Thus, it is important to have some idea of the properties and composition of the waste. The containers must have adequate wall thickness to withstand handling during sample collection and transport to the laboratory. Containers with wide mouths are desirable to facilitate transfer of samples from samplers to containers. Also, the containers must be large enough to contain the required volume of sample or the entire volume of a sample contained in samplers.

Plastic and glass containers are generally used for collecting and storing of hazardous waste samples. Commonly available plastic containers are made of high-density or linear polyethylene (LPE), conventional polyethylene, polypropylene, polycarbonate, teflon FEP (flourinated ethylene propylene), polyvinyl chloride (PVC), or polymethylpentene. Teflon FEP is almost universally usuable due to its chemical inertness and resistance to breakage. However, its high cost severely limits its use. LPE, on the other hand, offers the best combination of chemical resistance and low cost when inorganic wastes are involved.

Glass containers are relatively inert to most chemicals and can be used to collect and store almost all hazardous waste samples except those that contain strong alkali and hydroflouric acid. Soda glass bottles are suggested due to their low cost and ready availability. Borosilicate glass containers, such as Pyrex and Corex, have advantages relative to inertness and resistance to breakage respectively but are expensive and not always readily available. Glass containers are generally more fragile and much heavier than plastic containers. Glass or FEP containers must be used for waste samples that will be analyzed for organic compounds.

The containers must have tight, screw-type lids. Plastic bottles are usually provided with screw caps made of the same material as the bottles. Buttress threads are recommended. Cap liners are not usually required for plastic containers. Teflon cap liners should be used with glass containers supplied with rigid plastic screw caps. These caps are usually provided with waxed paper liners. Other liners that may be suitable are polyethylene, polypropylene, neoprene, and teflon FEP plastics. Teflon liners may be purchased from plastic specialty supply houses (e.g., Scientific Specialties Service, Inc., P.O. Box 352, Randallstown, Maryland 21133).

APPENDIX C-4

RANDOM SAMPLING PROCEDURES

RANDOM SAMPLING METHODOLOGY

Random Number Table

03	47	43	73	86	36	96	47	36	61	46	98	63	71	62
97	74	24	67	62	42	81	14	57	20	42	53	32	37	32
16	76	62	27	66	56	50	26	71	07	32	90	79	78	53
12	56	85	99	26	96	96	68	27	31	05	03	72	93	15
55	59	56	35	64	38	54	82	46	22	31	62	43	09	90
16	22	77	94	39	49	54	43	54	82	17	37	93	23	78
84	42	17	53	31	57	24	55	06	88	77	04	74	47	67
63	01	63	78	59	16	95	55	67	19	98	10	50	71	75
33	21	12	34	29	78	64	56	07	82	52	42	07	44	38
57	60	86	32	44	09	47	27	96	54	49	17	46	09	62
18	18	07	92	46	44	17	16	58	09	79	83	86	19	62
26	62	38	97	75	84	16	07	44	99	83	11	46	32	24
23	42	40	64	74	82	97	77	77	81	07	45	32	14	08
52	36	28	19	95	50	92	26	11	97	00	56	76	31	38
37	85	94	35	12	83	39	50	08	30	42	34	07	96	88
70	29	17	12	13	40	33	20	38	26	13	89	51	03	74
56	62	18	37	35	96	83	50	87	75	97	12	25	93	47
99	49	57	22	77	88	42	95	45	72	16	64	36	16	00
16	08	15	04	72	33	27	14	34	09	45	59	34	68	49
31	16	93	32	43	50	27	89	87	19	20	15	37	00	49

HOW TO USE THE TABLE OF RANDOM NUMBERS

1. Based on available information, segregate the containers (i.e., drums, sacks, etc.) according to waste types.

2. Number the containers containing the same waste types consecutively, starting from 01.

3. Decide on how many samples you wish to take. This number is usually determined by the objective of the sampling. SCI normally samples 10% of the waste containers holding each waste type. Hence if there were 50 drums containing the same type of waste, 5 drums have to be sampled.

4. Using the set of random numbers provided in the Random Number Table, choose any number as a starting point.

5. From this number, go down the column, then to the next column to the right, or go in any predetermined direction until you have selected five numbers between 01 and 20, with no repetitions. Larger numbers are ineligible.

   Example: If you were to choose 19 as the starting point on column four, the next eligible numbers as you go down this column are 12 and 04. So far you have chosen only three eligible numbers. Proceed to the next column to the right. Going down and starting from the top of this column, the next eligible numbers are 12 and 13. But 12 is already chosen. Proceeding to the sixth column, the next eligible number is 16. Your five random numbers, therefore, are 19, 12, 04, 13 and 16. Thus the drums with the corresponding numbers have to be sampled.

APPENDIX C.5-1

WASTE MOVEMENT
FORM

Sample
Waste Movement Form

FROM _____

TO _____

VOLUME _____ CT _____ GAL
Liquid Solid Sludge

DATE _____

ISSUED BY _____ TIME _____

TRANSFER BY _____

COMPLETED BY (TIME) _____

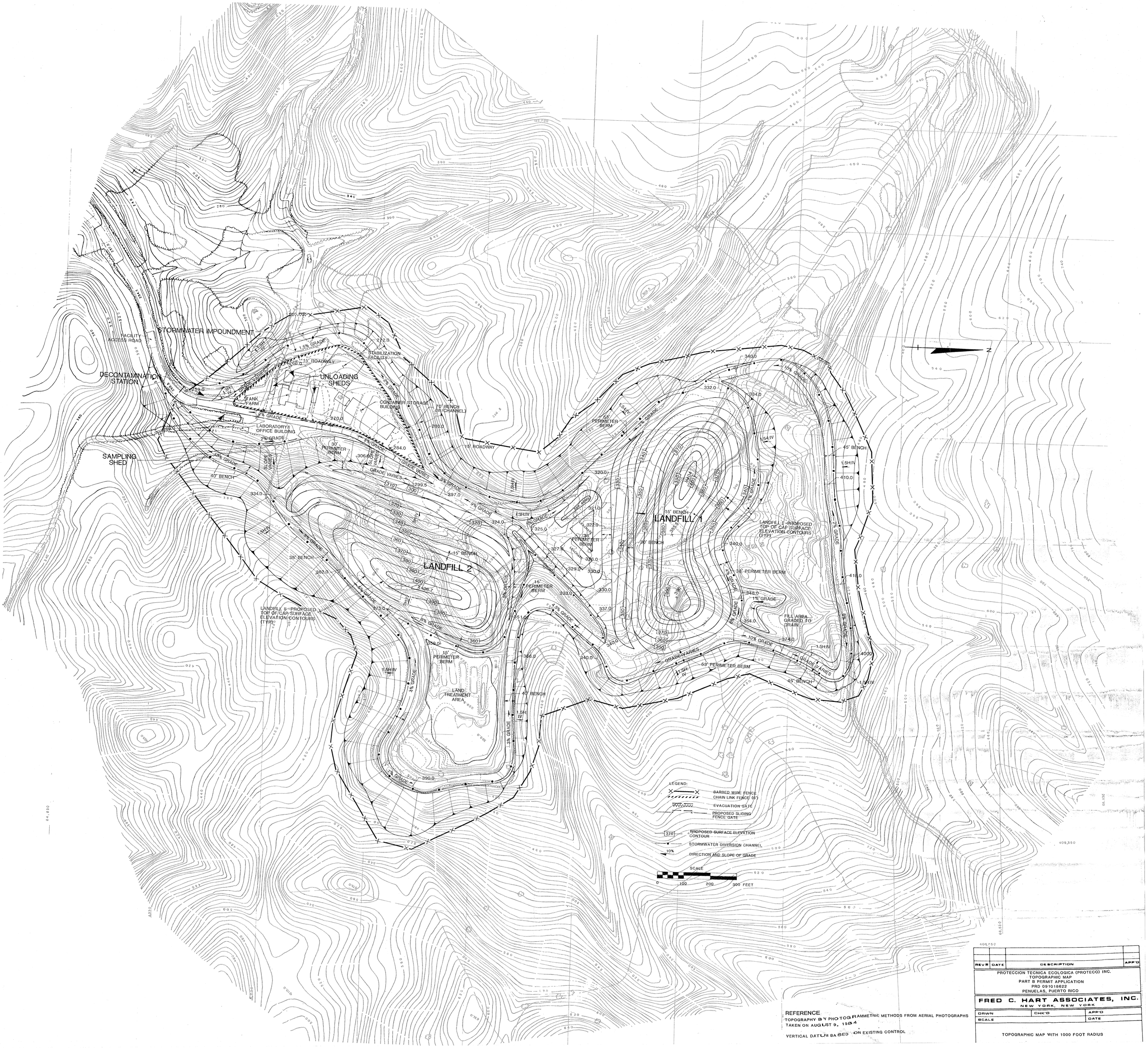
FORM RECEIVED IN LAB BY _____ TIME _____

INVENTORY VERIFICATION BY _____ TIME _____

(0062c-2)

	TANK READING	CONTAINERS (PIECE COUNT)
INITIAL		
FINAL		

SPECIAL HANDLING PROCEDURES



REFERENCE
TOPOGRAPHY BY PHOTOGRAMMETRIC METHODS FROM AERIAL PHOTOGRAPHS
TAKEN ON AUGUST 9, 1984
VERTICAL DATUM BASED ON EXISTING CONTROL

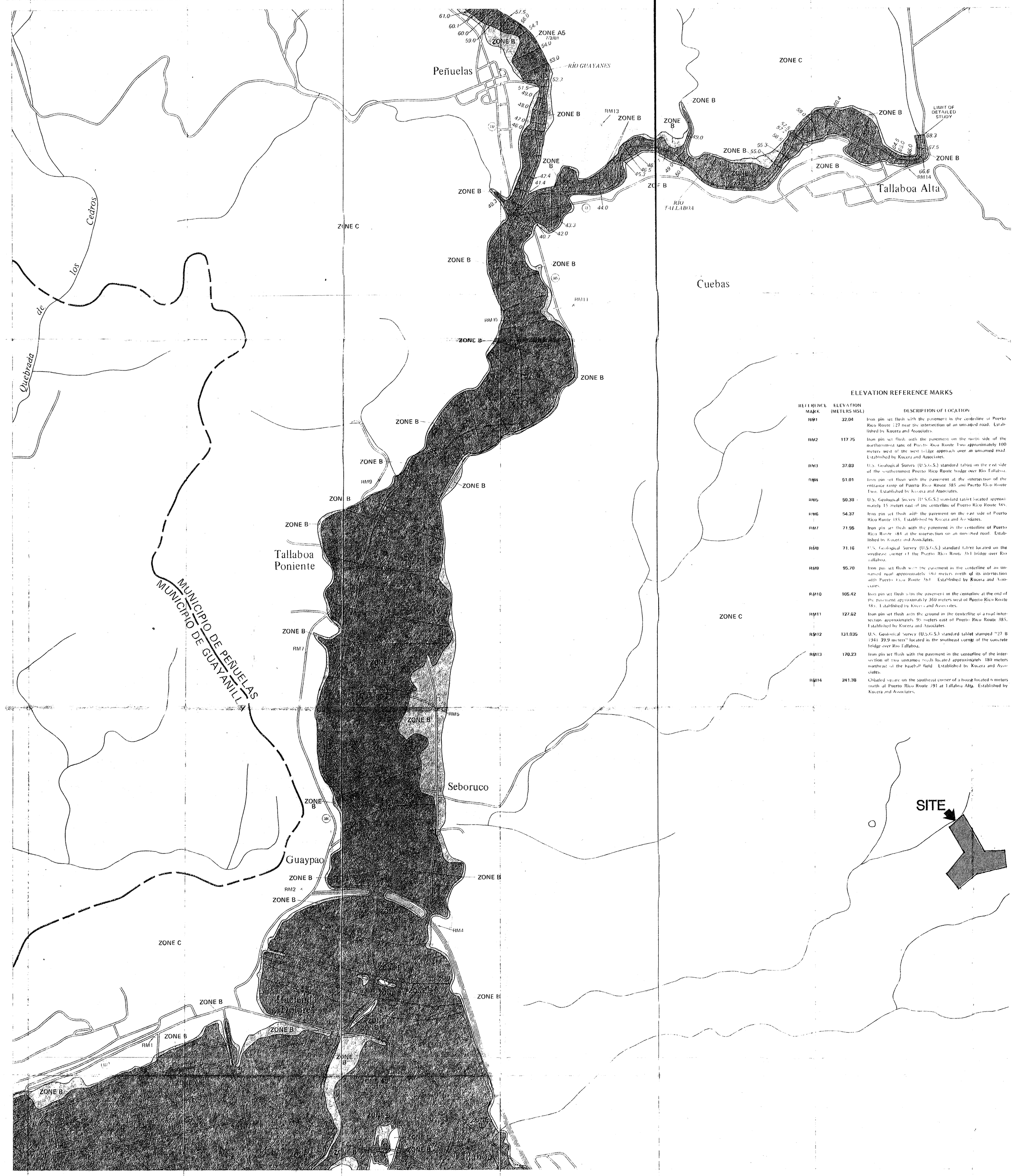
REV #	DATE	DESCRIPTION	APP'D

PROTECCION TECNICA ECOLOGICA (PROTECO) INC.
TOPOGRAPHIC MAP
PART B PERMIT APPLICATION
PRD 091018622
PENUELAS, PUERTO RICO

FRED C. HART ASSOCIATES, INC.
NEW YORK, NEW YORK

DRWN	CHK'D	APP'D
SCALE		DATE

TOPOGRAPHIC MAP WITH 1000 FOOT RADIUS



ELEVATION REFERENCE MARKS

REFERENCE MARK	ELEVATION (METERS MSL)	DESCRIPTION OF LOCATION
RM1	32.04	Iron pin set flush with the pavement in the centerline of Puerto Rico Route 127 near the intersection of an unnamed road. Established by Kucera and Associates.
RM2	117.75	Iron pin set flush with the pavement on the north side of the northernmost lane of Puerto Rico Route Two approximately 100 meters west of the west bridge approach over an unnamed road. Established by Kucera and Associates.
RM3	37.03	U.S. Geological Survey (U.S.G.S.) standard tablet on the east side of the southernmost Puerto Rico Route bridge over Rio Tallaboa.
RM4	51.01	Iron pin set flush with the pavement at the intersection of the entrance ramp of Puerto Rico Route 385 and Puerto Rico Route Two. Established by Kucera and Associates.
RM5	50.38	U.S. Geological Survey (U.S.G.S.) standard tablet located approximately 15 meters east of the centerline of Puerto Rico Route 385.
RM6	54.37	Iron pin set flush with the pavement on the east side of Puerto Rico Route 385. Established by Kucera and Associates.
RM7	71.95	Iron pin set flush with the pavement in the centerline of Puerto Rico Route 384 at the intersection on an unnamed road. Established by Kucera and Associates.
RM8	71.16	U.S. Geological Survey (U.S.G.S.) standard tablet located on the southeast corner of the Puerto Rico Route 381 bridge over Rio Tallaboa.
RM9	95.70	Iron pin set flush with the pavement in the centerline of an unnamed road approximately 380 meters north of its intersection with Puerto Rico Route 384. Established by Kucera and Associates.
RM10	105.42	Iron pin set flush with the pavement in the centerline at the end of the pavement approximately 360 meters west of Puerto Rico Route 385. Established by Kucera and Associates.
RM11	127.62	Iron pin set flush with the ground in the centerline of a road intersection approximately 95 meters east of Puerto Rico Route 385. Established by Kucera and Associates.
RM12	131.035	U.S. Geological Survey (U.S.G.S.) standard tablet stamped "27 B 1941 39.9 meters" located in the southeast corner of the concrete bridge over Rio Tallaboa.
RM13	170.23	Iron pin set flush with the pavement in the centerline of the intersection of two unnamed roads located approximately 180 meters northeast of the baseball field. Established by Kucera and Associates.
RM14	241.38	Chiseled square on the southeast corner of a house located 6 meters north of Puerto Rico Route 391 at Tallaboa Alta. Established by Kucera and Associates.

KEY TO MAP

500-Year Flood Boundary
100-Year Flood Boundary
Zone Designations* With Date of Identification e.g., 12/2/74
100-Year Flood Boundary
500-Year Flood Boundary

Base Flood Elevation Line With Elevation In Meters** — 153

Base Flood Elevation Line Where Uniform Within Zone** — (EL 387)

Elevation Reference Mark — RM7

**Referenced to Mean Sea Level

*EXPLANATION OF ZONE DESIGNATIONS

ZONE	EXPLANATION
A	Areas of 100-year flood; base flood elevations and flood hazard factors not determined.
AO	Areas of 100-year shallow flooding where depths are between 0.3 and 1.0 meter; average depths of inundation are shown, but no flood hazard factors are determined.
AH	Areas of 100-year shallow flooding where depths are between 0.3 and 1.0 meter; base flood elevations are shown, but no flood hazard factors are determined.
A1-A30	Areas of 100-year flood; base flood elevations and flood hazard factors determined.
A99	Areas of 100-year flood to be protected by flood protection system under construction; base flood elevations and flood hazard factors not determined.
B	Areas between limits of the 100-year flood and 500-year flood; or certain areas subject to 100-year flooding with average depths less than 0.3 meter or where the contributing drainage area is less than 2.6 square kilometers, or areas protected by levees from the base flood. (Medium shading)
C	Areas of minimal flooding. (No shading)
D	Areas of undetermined, but possible, flood hazards.
V	Areas of 100-year coastal flood with velocity (wave action); base flood elevations and flood hazard factors not determined.
V1-V30	Areas of 100-year coastal flood with velocity (wave action); base flood elevations and flood hazard factors determined.

N

APPROXIMATE SCALE
1" = 10000

1/2 1/4 0 1/2 Mile

500 0 500 1000 1500 2000 2500 3000 3000 Feet

.5 .25 0 .5 Kilometer

This map was prepared from U.S. Geological Survey 7.5-minute quadrangle maps and selected data.

This map was prepared to support municipal flood plain management regulations; it may not show all areas subject to flooding in this community or all drainage features outside special flood hazard areas.

Location areas not in the special flood hazard areas (zones A and V) may be protected by flood control structures.

For information on flood insurance is available in this community, contact your insurance agent or call the National Flood Insurance Program, at (800) 638-6620, or (800) 424-8872.

For adjoining map panels, see separately printed Index for Map Panels.

INITIAL IDENTIFICATION:
AUGUST 1, 1978

FLOOD INSURANCE RATE MAP EFFECTIVE:
AUGUST 1, 1978

FLOOD INSURANCE RATE MAP REVISIONS:
Map revised July 4, 1981 to add detailed flood hazard areas and elevations, and to change scale.

FIGURE B-2

FLOOD INSURANCE RATE MAP

COMMONWEALTH OF PUERTO RICO

HART

Fred C. Hart Associates, Inc.