



United States
Environmental Protection
Agency

FINAL UPDATE IIIB AND METHOD 9071B OF FINAL UPDATE IIIA

**Test Methods for Evaluating Solid Waste
Physical/Chemical Methods
SW-846**

Office of Solid Waste and
Emergency Response (5102G)
EPA-SW-846-03-03B
www.epa.gov
November 2004

**FINAL UPDATE IIIB
AND METHOD 9071B FROM FINAL UPDATE IIIA
TEST METHODS FOR EVALUATING SOLID WASTE
PHYSICAL/CHEMICAL METHODS
(SW-846) THIRD EDITION**

**THIS PACKET CONTAINS MATERIAL
FOR INCLUSION IN THE EPA PUBLICATION SW-846**

Contents:

1. **Cover sheet.** (What you are currently reading)
2. **Instructions.** This section explains how this package relates to the rest of SW-846.
3. **Status Tables.** The "Status Tables" document includes two tables. One table is a sequentially numbered listing of all SW-846 methods and their current status. The other table lists the status of all other documents in SW-846 (e.g., the Disclaimer, the chapters).
4. **Table of Contents.** The Table of Contents (dated November 2004) lists all of the methods (as updated by Third Edition Final Updates I, II, IIA, IIB, III, and IIIA and IIIB) in the order of appearance in the manual. It does not reflect Draft Update IVA or IVB methods because those methods are not yet finalized by the U.S. EPA.
5. **Chapter Five and Methods 9010C, 9012B, 9060A, 9070A, and 9071B (9071B is unchanged from Final Update IIIA).**
6. **Chapter Six and Methods 9045D and 9095B.**
7. **Chapter Seven**
8. **Chapter Eight and Methods 1010A, 1020B, 9040C, 1110A, and 1310B**

INSTRUCTIONS

SW-846, a methods manual, is a "living" document that changes when new data and advances in analytical techniques are incorporated into the manual as new or revised methods. To date, the Agency has formally issued Final Updates I, II, IIA, IIB, III, and IIIA, and Draft Updates IVA and IVB. (Currently, the methods and chapters of Draft Update IVB are not available from GPO, but are available on-line at www.epa.gov/SW-846/ or on paper from NTIS, (800) 553-6847.) This package contains Final Update IIIB methods and chapters with a footer date of November 2004. In addition, Method 9071B of Final Update IIIA with a footer date of April 1998 is included in this package because it was not distributed by the GPO at the time of its promulgation. Previously, it was only available through NTIS or at the SW-846 internet site.

These instructions describe how to get your basic manual up-to-date and what to do with your update package. A number of SW-846 update packages have been released to the public since the original Third Edition was released. In general, final updates should always be incorporated into SW-846 in chronological order (e.g., Update I should be incorporated before Update II).

If you got your package from GPO, the following definitions are provided as they relate to the text to follow regarding how to put together a GPO subscription to SW-846:

Previous subscribers are individuals that received copies of the Third Edition and other SW-846 updates (including proposed updates) in the past and just received this Final Update IIIB package in the mail.

New subscribers are individuals that recently (e.g., 6-8 weeks) placed an order with the GPO and received new copies of the 4 (four) volume set of the Third Edition, a copy of Final Update I, a copy of Final Update II/IIA, a copy of Final Update IIB, a copy of Final Update III, a copy of Draft Update IVA, and a copy of this package.

INCORPORATING FINAL UPDATE IIIB
AND METHOD 9071B (FROM FINAL UPDATE IIIA) IN YOUR MANUAL

This package is printed on white paper and all SW-846 chapters and methods finalized and updated as part of Update IIIB have the date of "November 2004" in the lower right hand corner. Method 9071B was last finalized and updated as part of Update IIIA and thus has the date of April 1998 in its lower right hand corner. You may have gotten this package from the National Technical Information Service (NTIS) or the Government Printing Office (GPO). Please visit the EPA OSW Methods Team website at <http://www.epa.gov/SW-846/> for information on other ways to obtain SW-846 methods and updates.

Please note that, if you wish to keep all of the final, proposed, and draft updates together, you will find that the binders provided to you as part of a GPO subscription are not large enough to contain all of the material. In that case, you can add other binders of your choice. The instructions below can be followed as appropriate if you get your SW-846 from other sources besides GPO.

NEW GPO SUBSCRIBERS - If you are a new subscriber, you should perform the following tasks before addressing this package (those who obtained their manual from other sources, may also find this information useful in putting together SW-846). You will need to use the detailed instructions found in the Final Update III package.

- Following the instructions found in your Final Update III package (starting at page "Instructions - 2" of that update package), place the original Third Edition of SW-846 (September 1986) in the properly labeled four 3-ring notebooks, and then incorporate Final Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), and III (December 1996).
- Either incorporate Draft Update IVA in the manual (without removing any white pages), or keep the colored draft update in a separate binder of your choice. Draft Update IVB is only available at the SW-846 internet site, at <http://www.epa.gov/SW-846/>, and may be printed and added to the manual in the same manner.
- Incorporate this package [Method 9071B of Final Update IIIA, dated April 1998, and Final Update IIIB, dated November 2004)] into the manual and remove any previous versions of its chapters and methods as appropriate.

PREVIOUS GPO SUBSCRIBERS - If you are a previous subscriber, it is important to establish exactly what is currently contained in your manual before addressing this package. If your manual is to be updated with only the latest final versions of the methods and chapters, the only white pages in the document should be dated September 1986 (Third Edition), July 1992 (Final Update I), August 1993 (Final Update IIA), September 1994 (Final Update II), January 1995 (Final Update IIB), December 1996 (Final Update III), and April 1998 (if you have added Final Update IIIA from NTIS or the EPA OSW Methods web site). Remove (and recycle or archive) any white

pages from your manual that have other dates -- unless you have printed, on white paper, other methods (e.g., Draft Update IVB methods) from the EPA web site and would like to retain them in your manual copy.

There may also be colored pages (e.g., pink pages for Proposed Update III) inserted in the manual. Remove all yellow, blue, green, or pink pages from the manual. These colored pages represent proposed versions of methods and chapters that have since been finalized. You may chose to keep your copies of colored versions in separate binders.

Finally, incorporate this package into the manual, removing any previous versions of its methods and chapters. (You may choose to keep the previous versions in a separate binder.)

UPDATE HISTORY OF SW-846

Package	Date	Paper Color	Status of Package
Third Edition	September 1986	White	Finalized (Promulgated)
Proposed Update I	December 1987	Green	Obsolete
Final Update I	November 1990	White	Obsolete
Proposed Update II	November 1990	Blue	Obsolete
Final Update I	July 1992	White	Finalized (Promulgated)
Proposed Update II	November 1992	Yellow	Obsolete
Proposed Update IIA	October 1992	White	Obsolete
Final Update IIA (Method 4010, included with Final Update II.)	August 1993	White	Finalized (Promulgated)
Final Update II	September 1994	White	Finalized (Promulgated)
Final Update IIB	January 1995	White	Finalized (Promulgated)
Proposed Update III	January 1995	Pink	Obsolete
Final Update III	December 1996	White	Finalized (Promulgated)
Draft Update IVA	January 1998	Salmon	Draft
Final Update IIIA	April 1998	White	Finalized (Promulgated)
Draft Update IVB	Nov. 2000	Get from www.epa.gov/ SW-846	Draft
Proposed Update IIIB	August 2002	Buff	Obsolete
Final Update IIIB	November 2004	White	Finalized (Promulgated) The GPO package also includes Method 9071B from Update IIIA

ASSISTANCE

After reading these instructions, if you need help due to difficulties understanding the status of the package or have technical questions regarding the methods, you may telephone the **Methods Information Communication Exchange (MICE)** at **703-676-4690** or send an E-mail to: mice@saic.com.

If you have questions concerning your **SW-846 U.S. Government Printing Office (GPO)** subscription, you should telephone the GPO at **202-512-1806**. If you did not purchase your SW-846 from the GPO, the GPO will not be able to help you.

**STATUS TABLES FOR
SW-846, THIRD EDITION**

ADDRESSES:

**FINAL UPDATES I, II, IIA, IIB, III, IIIA, AND IIIB
DRAFT UPDATES IVA AND IVB
OTHER METHODS AT THE OSW METHODS WEB SITE**

NOVEMBER 2004

HOW TO USE THIS DOCUMENT

This document provides historical information regarding EPA-published draft, proposed, and final SW-846 methods and chapters. It contains two status tables, namely; the "SW-846 Method Status Table," which is a listing of SW-846 methods; and the "Status Table for SW-846 Chapter Text and Other Documents," which lists all other documents in SW-846.

Use the "SW-846 Method Status Table" as a reference guide to identify the historical and latest versions of SW-846 methods. Methods in this status table are listed sequentially by method number. The column showing "Other Methods" includes those methods that appear as new SW-846 methods at EPA's Office of Solid Waste Methods Team internet site, <http://www.epa.gov/SW-846/>. An integrated version of the manual is also available at the Methods Team internet site.

Use the "Status Table for SW-846 Chapter Text and Other Documents" as a reference guide to identify the historical and latest versions of chapters and other SW-846 documents (e.g., the Disclaimer).

Previous versions of the "SW-846 Method Status Table" included a column for "Current Promulgated Method." The November 2004 version of that table does not contain that column because, with the publication of the final Methods Innovation Rule, SW-846 and its methods are no longer required in general by any RCRA regulation. See 40 CFR 260.11(a)(11) for a listing of those SW-846 methods that may be still required by the RCRA regulations for the analysis of method-defined parameters.

Do not use a status table as a guide for putting together a paper version of SW-846. Refer to the "Table of Contents" of the update for the order in which chapters and methods should appear in SW-846.

SW-846 METHOD STATUS TABLE
November 2004

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
0010	—	—	—	—	—	—	—	Modified Method 5 Sampling Train
—	—	—	0011 (Up. III)	—	—	—	—	Sampling for Selected Aldehyde and Ketone Emissions from Stationary Sources
0020	—	—	—	—	—	—	—	Source Assessment Sampling System (SASS)
—	—	—	0023A (Up. III) Revision of Method 23, 40 CFR Part 60	—	—	—	—	Sampling Method for Polychlorinated Dibenzo- <i>p</i> -Dioxins and Polychlorinated Dibenzofuran Emissions from Stationary Sources
—	—	—	—	—	—	25D Referral	—	Determination of the Volatile Organic Content of Waste Samples
—	—	—	—	—	—	25E Referral	—	Determination of Vapor Phase Organic Concentration in Waste Samples
0030	—	—	—	—	—	—	—	Volatile Organic Sampling Train
—	—	—	0031 (Up. III)	—	—	—	—	Sampling Method for Volatile Organic Compounds (SMVOC)
—	—	—	0040 (Up. III)	—	—	—	—	Sampling of Principal Organic Hazardous Constituents from Combustion Sources Using Tedlar® Bags

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
—	—	—	0050 (Up. III)	—	—	—	—	Isokinetic HCl/Cl ₂ Emission Sampling Train
—	—	—	0051 (Up. III)	—	—	—	—	Midget Impinger HCl/Cl ₂ Emission Sampling Train
—	—	—	0060 (Up. III)	—	—	—	—	Determination of Metals in Stack Emissions
—	—	—	0061 (Up. III)	—	—	—	—	Determination of Hexavalent Chromium Emissions from Stationary Sources
—	—	—	0100 (Up. III)	—	—	—	—	Sampling for Formaldehyde and Other Carbonyl Compounds in Indoor Air
—	—	—	—	—	—	207-1 Referral	—	Sampling Method for Isocyanates
—	—	—	—	—	—	207-2 Referral	—	Analysis for Isocyanates by High Performance Liquid Chromatography (HPLC)
1010	—	—	—	1010A	—	—	—	Test Methods for Flash Point by Pensky-Martens Closed Cup Tester (Method text is a referral to ASTM Standard D 93-79 or Standard D 93-80)
1020	1020A	—	—	1020B	—	—	—	Standard Test Methods for Flash Point by Setaflash (Small Scale) Closed-cup Apparatus (Method text is a referral to ASTM Standard D 3278-78)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	--	--	1030 (Up. III)	--	--	--	--	Ignitability of Solids
--	--	--	--	--	--	1040	--	Test Method for Oxidizing Solids
--	--	--	--	--	--	1050	--	Test Methods to Determine Substances Likely to Spontaneously Combust
1110	--	--	--	1110A	--	--	--	Corrosivity Toward Steel
--	--	--	1120 (Up. III)	--	--	--	--	Dermal Corrosion
1310	1310A	--	--	1310B	--	--	--	Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test
--	1311	--	--	--	--	--	--	Toxicity Characteristic Leaching Procedure
--	--	1312 (Up. II)	--	--	--	--	--	Synthetic Precipitation Leaching Procedure
1320	--	--	--	--	--	--	--	Multiple Extraction Procedure
1330	1330A	--	--	--	--	--	--	Extraction Procedure for Oily Wastes
3005	3005A	--	--	--	--	--	--	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy
3010	3010A	--	--	--	--	--	--	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	--	3015 (Up. II)	--	--	3015A	--	--	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts
3020	3020A	--	--	--	--	--	--	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy
--	--	--	3031 (Up. III)	--	--	--	--	Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry
3040	--	--	3040A (Up. III)	--	--	--	--	Dissolution Procedure for Oils, Greases, or Waxes
3050	3050A	--	3050B (Up. III)	--	--	--	--	Acid Digestion of Sediments, Sludges, and Soils
--	--	3051 (Up. II)	--	--	3051A	--	--	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
--	--	--	3052 (Up. III)	--	--	--	--	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices
[3060, in the 2nd Ed.]	--	--	3060A (Up. III)	--	--	--	--	Alkaline Digestion for Hexavalent Chromium
3500	3500A	--	3500B (Up. III)	--	--	3500C	--	Organic Extraction and Sample Preparation
3510	3510A	3510B (Up. II)	3510C (Up. III)	--	--	--	--	Separatory Funnel Liquid-Liquid Extraction

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note The date in parenthesis is the date found at the bottom right-hand corner of the method

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	--	--	--	--	--	--	3511 (11/02)	Organic Compounds in Water by Microextraction
3520	3520A	3520B (Up II)	3520C (Up III)	--	--	--	--	Continuous Liquid-Liquid Extraction
--	--	--	3535 (Up III)	--	3535A	3535A (Replaces IVA version)	--	Solid-Phase Extraction (SPE)
3540	3540A	3540B (Up II)	3540C (Up III)	--	--	--	--	Soxhlet Extraction
--	--	3541 (Up II)	--	--	--	--	--	Automated Soxhlet Extraction
--	--	--	3542 (Up III)	--	--	--	--	Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train)
--	--	--	3545 (Up. III)	--	3545A	3545A (Replaces IVA version)	--	Pressurized Fluid Extraction (PFE)
--	--	--	--	--	--	3546	--	Microwave Extraction
3550	--	3550A (Up II)	3550B (Up III)	--	--	3550C	--	Ultrasonic Extraction

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note The date in parenthesis is the date found at the bottom right-hand corner of the method

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	--	--	3560 (Up. III)	--	--	--	--	Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons
--	--	--	3561 (Up. III)	--	--	--	--	Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons
--	--	--	--	--	3562	--	--	Supercritical Fluid Extraction of Polychlorinated Biphenyls (PCBs) and Organochlorine Pesticides
--	--	--	--	--	--	--	3570 (11/02)	Microscale Solvent Extraction (MSE)
3580	3580A	--	--	--	--	--	--	Waste Dilution
--	--	--	3585 (Up. III)	--	--	--	--	Waste Dilution for Volatile Organics
3600	3600A	3600B (Up. II)	3600C (Up. III)	--	--	--	--	Cleanup
3610	3610A	--	3610B (Up. III)	--	--	--	--	Alumina Cleanup
3611	3611A	--	3611B (Up. III)	--	--	--	--	Alumina Column Cleanup and Separation of Petroleum Wastes
3620	3620A	--	3620B (Up. III)	--	--	3620C	--	Florisil Cleanup
3630	3630A	3630B (Up. II)	3630C (Up. III)	--	--	--	--	Silica Gel Cleanup

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note The date in parenthesis is the date found at the bottom right-hand corner of the method

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
3640	--	3640A (Up. II)	--	--	--	--	--	Gel-Permeation Cleanup
3650	3650A	--	3650B (Up. III)	--	--	--	--	Acid-Base Partition Cleanup
3660	3660A	--	3660B (Up. III)	--	--	--	--	Sulfur Cleanup
--	--	3665 (Up. II)	3665A (Up. III)	--	--	--	--	Sulfuric Acid/Permanganate Cleanup
3810	--	--	--	--	Noticed for removal from SW-846	--	--	Headspace
--	--	--	--	--	--	3815	--	Screening Solid Samples for Volatile Organics
3820	--	--	--	--	--	--	--	Hexadecane Extraction and Screening of Purgeable Organics
--	--	--	4000 (Up. III)	--	--	--	--	Immunoassay
--	--	4010 (Up. IIA)	4010A (Up. III)	--	--	--	--	Screening for Pentachlorophenol by Immunoassay
--	--	--	4015 (Up. III)	--	--	--	--	Screening for 2,4-Dichlorophenoxyacetic Acid by Immunoassay

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	--	--	4020 (Up. III)	--	--	--	--	Screening for Polychlorinated Biphenyls by Immunoassay
--	--	--	--	--	--	--	4025 (10/02)	Screening for Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by Immunoassay
--	--	--	4030 (Up. III)	--	--	--	--	Soil Screening for Petroleum Hydrocarbons by Immunoassay
--	--	--	4035 (Up. III)	--	--	--	--	Soil Screening for Polynuclear Aromatic Hydrocarbons by Immunoassay
--	--	--	4040 (Up. III)	--	--	--	--	Soil Screening for Toxaphene by Immunoassay
--	--	--	4041 (Up. III)	--	--	--	--	Soil Screening for Chlordane by Immunoassay
--	--	--	4042 (Up. III)	--	--	--	--	Soil Screening for DDT by Immunoassay
--	--	--	--	--	--	--	4025 (10/02)	Screening for Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by Immunoassay
--	--	--	4050 (Up. III)	--	--	--	--	TNT Explosives in Soil by Immunoassay
--	--	--	4051 (Up. III)	--	--	--	--	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) in Soil by Immunoassay

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
—	—	—	—	—	—	4425	—	Screening Extracts of Environmental Samples for Planar Organic Compounds (PAHs, PCBs, PCDDs/PCDFs) by a Reporter Gene on a Human Cell Line
—	—	—	—	—	4500	—	—	Mercury in Soil by Immunoassay
—	—	—	—	—	4670	—	—	Triazine Herbicides as Atrazine in Water by Quantitative Immunoassay
—	—	—	5000 (Up. III)	—	—	—	—	Sample Preparation for Volatile Organic Compounds
—	—	—	5021 (Up. III)	—	—	—	5021A (6/03)	5021A: Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis
5030	5030A	—	5030B (Up. III)	—	—	—	5030C (5/03)	Purge-and-Trap for Aqueous Samples
—	—	—	5031 (Up. III)	—	—	—	—	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation
—	—	—	5032 (Up. III)	—	—	—	—	Volatile Organic Compounds by Vacuum Distillation
—	—	—	5035 (Up. III)	—	—	—	5035A (7/02)	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
5040	--	5040A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Analysis of Sorbent Cartridges from Volatile Organic Sampling Train (VOST): Gas Chromatography/Mass Spectrometry Technique
--	--	5041 (Up. II)	5041A (Up. III)	--	--	--	--	Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST)
--	--	5050 (Up. II)	--	--	--	--	--	Bomb Preparation Method for Solid Waste
6010	6010A	--	6010B (Up. III)	--	--	6010C	--	Inductively Coupled Plasma-Atomic Emission Spectrometry
--	--	6020 (Up. II)	--	--	6020A	--	--	Inductively Coupled Plasma - Mass Spectrometry
--	--	--	--	--	6200	--	--	Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment
--	--	--	--	--	6500	--	--	Dissolved Inorganic Anions in Aqueous Matrices by Capillary Ion Electrophoresis
--	--	--	--	--	6800	--	--	Elemental and Speciated Isotope Dilution Mass Spectrometry
7000	7000A	--	--	--	7000B	--	--	7000B (Draft Up. IVA): Flame Atomic Absorption Spectrophotometry
--	--	--	--	--	7010	--	--	Graphite Furnace Atomic Absorption Spectrophotometry

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
7020	--	--	--	--	Noticed for removal from SW-846	--	--	Aluminum (Atomic Absorption, Direct Aspiration)
7040	--	--	--	--	Noticed for removal from SW-846	--	--	Antimony (Atomic Absorption, Direct Aspiration)
7041	--	--	--	--	Noticed for removal from SW-846	--	--	Antimony (Atomic Absorption, Furnace Technique)
7060	--	7060A (Up. II)	--	--	Noticed for removal from SW-846	--	--	Arsenic (Atomic Absorption, Furnace Technique)
7061	7061A	--	--	--	--	--	--	Arsenic (Atomic Absorption, Gaseous Hydride)
--	--	7062 (Up. II)	--	--	--	--	--	Antimony and Arsenic (Atomic Absorption, Borohydride Reduction)
--	--	--	7063 (Up. III)	--	--	--	--	Arsenic in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
7080	—	7080A (Up. II)	—	—	Noticed for removal from SW-846	—	—	Barium (Atomic Absorption, Direct Aspiration)
—	7081	—	—	—	Noticed for removal from SW-846	—	—	Barium (Atomic Absorption, Furnace Technique)
7090	—	—	—	—	Noticed for removal from SW-846	—	—	Beryllium (Atomic Absorption, Direct Aspiration)
7091	—	—	—	—	Noticed for removal from SW-846	—	—	Beryllium (Atomic Absorption, Furnace Technique)
7130	—	—	—	—	Noticed for removal from SW-846	—	—	Cadmium (Atomic Absorption, Direct Aspiration)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
7131	--	7131A (Up II)	--	--	Noticed for removal from SW-846	--	--	Cadmium (Atomic Absorption, Furnace Technique)
7140	--	--	--	--	Noticed for removal from SW-846	--	--	Calcium (Atomic Absorption, Direct Aspiration)
7190	--	--	--	--	Noticed for removal from SW-846	--	--	Chromium (Atomic Absorption, Direct Aspiration)
7191	--	--	--	--	Noticed for removal from SW-846	--	--	Chromium (Atomic Absorption, Furnace Technique)
7195	--	--	--	--	--	--	--	Chromium, Hexavalent (Coprecipitation)
7196	7196A	--	--	--	--	--	--	Chromium, Hexavalent (Colorimetric)
7197	--	--	--	--	--	--	--	Chromium, Hexavalent (Chelation/Extraction)
7198	--	--	--	--	--	--	--	Chromium, Hexavalent (Differential Pulse Polarography)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
—	—	—	7199 (Up. III)	—	—	—	—	Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography
7200	—	—	—	—	Noticed for removal from SW-846	—	—	Cobalt (Atomic Absorption, Direct Aspiration)
7201	—	—	—	—	Noticed for removal from SW-846	—	—	Cobalt (Atomic Absorption, Furnace Technique)
7210	—	—	—	—	Noticed for removal from SW-846	—	—	Copper (Atomic Absorption, Direct Aspiration)
—	7211	—	—	—	Noticed for removal from SW-846	—	—	Copper (Atomic Absorption, Furnace Technique)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
7380	--	--	--	--	Noticed for removal from SW-846	--	--	Iron (Atomic Absorption, Direct Aspiration)
--	7381	--	--	--	Noticed for removal from SW-846	--	--	Iron (Atomic Absorption, Furnace Technique)
7420	--	--	--	--	Noticed for removal from SW-846	--	--	Lead (Atomic Absorption, Direct Aspiration)
7421	--	--	--	--	Noticed for removal from SW-846	--	--	Lead (Atomic Absorption, Furnace Technique)
--	7430	--	--	--	Noticed for removal from SW-846	--	--	Lithium (Atomic Absorption, Direct Aspiration)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note. The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
7450	--	--	--	--	Noticed for removal from SW-846	--	--	Magnesium (Atomic Absorption, Direct Aspiration)
7460	--	--	--	--	Noticed for removal from SW-846	--	--	Manganese (Atomic Absorption, Direct Aspiration)
--	7461	--	--	--	Noticed for removal from SW-846	--	--	Manganese (Atomic Absorption, Furnace Technique)
7470	--	7470A (Up II)	--	--	--	--	--	Mercury in Liquid Waste (Manual Cold-Vapor Technique)
7471	--	7471A (Up II)	--	--	7471B	--	--	Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)
--	--	--	7472 (Up III)	--	--	--	--	Mercury in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)
--	--	--	--	--	7473	--	--	Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
—	—	—	—	—	7474	—	—	Mercury in Sediment and Tissue Samples by Atomic Fluorescence Spectrometry
7480	—	—	—	—	Noticed for removal from SW-846	—	—	Molybdenum (Atomic Absorption, Direct Aspiration)
7481	—	—	—	—	Noticed for removal from SW-846	—	—	Molybdenum (Atomic Absorption, Furnace Technique)
7520	—	—	—	—	Noticed for removal from SW-846	—	—	Nickel (Atomic Absorption, Direct Aspiration)
—	—	—	7521 (Up. III)	—	Noticed for removal from SW-846	—	—	Nickel (Atomic Absorption, Furnace Method)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
7550	--	--	--	--	Noticed for removal from SW-846	--	--	Osmium (Atomic Absorption, Direct Aspiration)
--	--	--	7580 (Up. III)	--	--	--	--	White Phosphorus (P ₄) by Solvent Extraction and Gas Chromatography
7610	--	--	--	--	Noticed for removal from SW-846	--	--	Potassium (Atomic Absorption, Direct Aspiration)
7740	--	--	--	--	Noticed for removal from SW-846	--	--	Selenium (Atomic Absorption, Furnace Technique)
7741	--	7741A (Up. II)	--	--	--	--	--	Selenium (Atomic Absorption, Gaseous Hydride)
--	--	7742 (Up. II)	--	--	--	--	--	Selenium (Atomic Absorption, Borohydride Reduction)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
7760	7760A	--	--	--	Noticed for removal from SW-846	--	--	Silver (Atomic Absorption, Direct Aspiration)
--	7761	--	--	--	Noticed for removal from SW-846	--	--	Silver (Atomic Absorption, Furnace Technique)
7770	--	--	--	--	Noticed for removal from SW-846	--	--	Sodium (Atomic Absorption, Direct Aspiration)
--	7780	--	--	--	Noticed for removal from SW-846	--	--	Strontium (Atomic Absorption, Direct Aspiration)
7840	--	--	--	--	Noticed for removal from SW-846	--	--	Thallium (Atomic Absorption, Direct Aspiration)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
7841	—	—	—	—	Noticed for removal from SW-846	—	—	Thallium (Atomic Absorption, Furnace Technique)
7870	—	—	—	—	Noticed for removal from SW-846	—	—	Tin (Atomic Absorption, Direct Aspiration)
7910	—	—	—	—	Noticed for removal from SW-846	—	—	Vanadium (Atomic Absorption, Direct Aspiration)
7911	—	—	—	—	Noticed for removal from SW-846	—	—	Vanadium (Atomic Absorption, Furnace Technique)
7950	—	—	—	—	Noticed for removal from SW-846	—	—	Zinc (Atomic Absorption, Direct Aspiration)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	7951	--	--	--	Noticed for removal from SW-846	--	--	Zinc (Atomic Absorption, Furnace Technique)
8000	8000A	--	8000B (Up. III)	--	--	--	8000C (3/03)	Determinative Chromatographic Separations
8010	8010A	8010B (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Halogenated Volatile Organics by Gas Chromatography
--	8011	--	--	--	--	--	--	1,2-Dibromoethane and 1,2-Dibromo-3- chloropropane by Microextraction and Gas Chromatography
8015	8015A	--	8015B	--	--	8015C	8015D (6/03)	Nonhalogenated Organics Using GC/FID
8020	--	8020A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Aromatic Volatile Organics by Gas Chromatography
--	8021	8021A (Up. II)	8021B (Up. III)	--	--	--	--	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
8030	8030A	--	Deleted from SW-846 (Up. III)	--	--	--	--	Acrolein and Acrylonitrile by Gas Chromatography
--	--	8031 (Up. II)	--	--	--	--	--	Acrylonitrile by Gas Chromatography
--	--	8032 (Up. II)	8032A (Up. III)	--	--	--	--	Acrylamide by Gas Chromatography
--	--	--	8033 (Up. III)	--	--	--	--	Acetonitrile by Gas Chromatography with Nitrogen-Phosphorus Detection
8040	8040A	--	Deleted from SW-846 (Up. III)	--	--	--	--	Phenols by Gas Chromatography
--	--	--	8041 (Up. III)	--	--	8041A	--	Phenols by Gas Chromatography
8060	--	--	Deleted from SW-846 (Up. III)	--	--	--	--	Phthalate Esters
--	--	8061 (Up. II)	8061A (Up. III)	--	--	--	--	Phthalate Esters by Gas Chromatography with Electron Capture Detection (GC/ECD)
--	8070	--	8070A (Up. III)	--	--	--	--	Nitrosamines by Gas Chromatography

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
8080	--	8080A (Up II)	Deleted from SW-846 (Up III)	--	--	--	--	Organochlorine Pesticides and Polychlorinated Biphenyls by Gas Chromatography
--	--	8081 (Up. II)	8081A (Up. III)	--	8081B	8081B (Replaces IVA version)	--	Organochlorine Pesticides by Gas Chromatography
--	--	--	8082 (Up. III)	--	8082A	8082A (Replaces IVA version)	--	Polychlorinated Biphenyls (PCBs) by Gas Chromatography
				--		8085	--	Compound-independent Elemental Quantitation of Pesticides by Gas Chromatography with Atomic Emission Detection (GC/AED)
8090	--	--	Deleted from SW-846 (Up. III)	--	--	--	--	Nitroaromatics and Cyclic Ketones
--	--	--	8091 (Up. III)	--	--	--	--	Nitroaromatics and Cyclic Ketones by Gas Chromatography
--	--	--	--	--	--	8095	--	Explosives by Gas Chromatography
8100	--	--	--	--	--	--	--	Polynuclear Aromatic Hydrocarbons

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	8110	--	Deleted from SW-846 (Up. III)	--	--	--	--	Haloethers by Gas Chromatography
--	--	--	8111 (Up. III)	--	--	--	--	Haloethers by Gas Chromatography
8120	--	8120A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Chlorinated Hydrocarbons by Gas Chromatography
--	--	8121 (Up. II)	--	--	--	--	--	Chlorinated Hydrocarbons by Gas Chromatography. Capillary Column Technique
--	--	--	8131 (Up. III)	--	--	--	--	Aniline and Selected Derivatives by Gas Chromatography
8140	--	--	Deleted from SW-846 (Up. III)	--	--	--	--	Organophosphorus Pesticides
--	8141	8141A (Up. II)	--	--	8141B	8141B (Replaces IVA version)	--	8141B. Organophosphorus Compounds by Gas Chromatography

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
8150	8150A	8150B (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Chlorinated Herbicides by Gas Chromatography
--	--	8151 (Up. II)	8151A (Up. III)	--	--	--	--	Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylaton Derivatization
8240	8240A	8240B (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
8250	--	8250A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
--	8260	8260A (Up. II)	8260B (Up. III)	--	--	--	--	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
--	--	--	--	--	--	8261	--	Volatile Organic Compounds by Vacuum Distillation in Combination with Gas Chromatography/Mass Spectrometry (VD/GC/MS)
--	--	--	--	--	--	--	8265 (3/02)	Volatile Organic Compounds in Water, Soil, Soil Gas and Air by Direct Sampling Ion Trap Mass Spectrometry (DSITMS)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
8270	8270A	8270B (Up. II)	8270C (Up. III)	--	8270D	--	--	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
--	--	8275 (Up. II)	8275A (Up. III)	--	--	--	--	Semivolatile Organic Compounds (PAHs and PCBs) in Soils/Sludges and Solid Wastes Using Thermal Extraction/Gas Chromatography/Mass Spectrometry (TE/GC/MS)
8280	--	--	8280A (Up. III)	--	8280B	--	--	8280B: Polychlorinated Dibenzo- <i>p</i> -Dioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)
--	--	8290 (Up. II)	--	--	8290A	--	--	Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)
8310	--	--	--	--	--	--	--	Polynuclear Aromatic Hydrocarbons
--	--	8315 (Up. II)	8315A (Up. III)	--	--	--	--	Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)
--	--	8316 (Up. II)	--	--	--	--	--	Acrylamide, Acrylonitrile and Acrolein by High Performance Liquid Chromatography (HPLC)
--	--	8318 (Up. II)	--	--	--	8318A	--	<i>N</i> -Methylcarbamates by High Performance Liquid Chromatography (HPLC)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
—	—	8321 (Up. II)	8321A (Up. III)	—	8321B	8321B (Replaces IVA version)	—	Solvent-Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection
—	—	—	—	—	—	—	8323 (1/03)	Determination of Organotin by Micro-liquid Chromatography-electrospray Ion Trap Mass Spectrometry
—	—	—	8325 (Up. III)	—	—	—	—	Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Particle Beam/Mass Spectrometry (HPLC/PB/MS)
—	—	8330 (Up. II)	—	—	8330A	—	—	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)
—	—	8331 (Up. II)	—	—	—	—	—	Tetrazene by Reverse Phase High Performance Liquid Chromatography (HPLC)
—	—	—	8332 (Up. III)	—	—	—	—	Nitroglycerine by High Performance Liquid Chromatography
—	—	8410 (Up. II)	—	—	—	—	—	Gas Chromatography/Fourier Transform Infrared (GC/FT-IR) Spectrometry for Semivolatile Organics: Capillary Column
—	—	—	8430 (Up. III)	—	—	—	—	Analysis of Bis(2-chloroethyl) Ether and Hydrolysis Products by Direct Aqueous Injection GC/FT-IR

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
—	—	—	8440 (Up. III)	—	—	—	—	Total Recoverable Petroleum Hydrocarbons by Infrared Spectrophotometry
				—		8510	—	Colorimetric Screening Procedure for RDX and HMX in Soil
—	—	—	8515 (Up. III)	—	—	—	—	Colorimetric Screening Method for Trinitrotoluene (TNT) in Soil
—	—	—	8520 (Up. III)	—	—	—	—	Continuous Measurement of Formaldehyde in Ambient Air
—	—	—	—	—	—	8535	—	Screening Procedure for Total Volatile Organic Halides in Water
—	—	—	—	—	—	8540	—	Pentachlorophenol by UV-induced Colorimetry
—	—	—	—	—	9000	—	—	Determination of Water in Waste Materials by Karl Fischer Titration
—	—	—	—	—	9001	—	—	Determination of Water in Waste Materials by Quantitative Calcium Hydride Reaction
9010	9010A	—	9010B (Up. III)	9010C	—	—	—	Total and Amenable Cyanide Distillation
9012	—	—	9012A (Up. III)	9012B	—	—	—	Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)
—	9013	—	—	—	—	—	—	Cyanide Extraction Procedure for Solids and Oils

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note. The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	--	--	9014 (Up. III)	--	--	--	--	Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide
--	--	--	--	--	--	--	9015 (9/04)	Metal Cyanide Complexes by Anion Exchange Chromatography and UV Detection
9020	9020A	9020B (Up. II)	--	--	--	--	--	Total Organic Halides (TOX)
--	9021	--	--	--	--	--	--	Purgeable Organic Halides (POX)
9022	--	--	--	--	--	--	--	Total Organic Halides (TOX) by Neutron Activation Analysis
--	--	--	9023 (Up. III)	--	--	--	--	Extractable Organic Halides (EOX) in Solids
9030	9030A	--	9030B (Up. III)	--	--	--	--	Acid-Soluble and Acid-Insoluble Sulfides. Distillation
--	9031	--	--	--	--	--	--	Extractable Sulfides
--	--	--	9034 (Up. III)	--	--	--	--	Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides
9035	--	--	--	--	--	--	--	Sulfate (Colorimetric, Automated, Chloranilate)
9036	--	--	--	--	--	--	--	Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)
9038	--	--	--	--	--	--	--	Sulfate (Turbidimetric)

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
9040	--	9040A (Up. II) 9040B (Up. IIB)	--	9040C	--	--	--	pH Electrometric Measurement
9041	9041A	--	--	--	--	--	--	pH Paper Method
9045	9045A	9045B (Up. II) 9045C (Up. IIB)	--	9045D	--	--	--	Soil and Waste pH
9050	--	--	9050A (Up. III)	--	--	--	--	Specific Conductance
--	--	9056 (Up. II)	--	--	--	9056A	--	Determination of Inorganic Anions by Ion Chromatography
--	--	--	9057 (Up. III)	--	--	--	--	Determination of Chloride from HCl/Cl ₂ Emission Sampling Train (Methods 0050 and 0051) by Anion Chromatography
				--		9058	--	Determination of Perchlorate Using Ion Chromatography with Chemical Suppression Conductivity Detection
9060	--	--	--	9060A	--	--	--	Total Organic Carbon
9065	--	--	--	--	--	--	--	Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)

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Note. The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
9066	--	--	--	--	--	--	--	Phenolics (Colorimetric, Automated 4-AAP with Distillation)
9067	--	--	--	--	--	--	--	Phenolics (Spectrophotometric, MBTH with Distillation)
9070	--	--	9070 Title and text replaced with referral to Method 1664 (Up. IIIA)	9070A (Suffix and title added)	--	--	--	n-Hexane Extractable Material (HEM) for Aqueous Samples (Method text is a referral to Method 1664: n-Hexane Extractable Material (HEM, Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry)
9071	--	9071A (Up. II)	9071B (Up. IIIA)	--	--	--	--	n-Hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples
--	--	--	--	--	9074	--	--	Turbidimetric Screening Method for Total Recoverable Petroleum Hydrocarbons in Soil
--	--	9075 (Up. II)	--	--	--	--	--	Test Method for Total Chlorine in New and Used Petroleum Products by X-Ray Fluorescence Spectrometry (XRF)
--	--	9076 (Up. II)	--	--	--	--	--	Test Method for Total Chlorine in New and Used Petroleum Products by Oxidative Combustion and Microcoulometry

SW-846 METHOD STATUS TABLE (11/04), CONTINUED

Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
--	--	9077 (Up. II)	--	--	--	--	--	Test Methods for Total Chlorine in New and Used Petroleum Products (Field Test Kit Methods)
--	--	--	9078 (Up. III)	--	--	--	--	Screening Test Method for Polychlorinated Biphenyls in Soil
--	--	--	9079 (Up. III)	--	--	--	--	Screening Test Method for Polychlorinated Biphenyls in Transformer Oil
9080	--	--	--	--	--	--	--	Cation-Exchange Capacity of Soils (Ammonium Acetate)
9081	--	--	--	--	--	--	--	Cation-Exchange Capacity of Soils (Sodium Acetate)
9090	9090A	--	--	--	--	--	--	Compatibility Test for Wastes and Membrane Liners
9095	--	--	9095A (Up. III)	9095B	--	--	--	Paint Filter Liquids Test
--	--	9096 (Up. II)	--	--	--	--	--	Liquid Release Test (LRT) Procedure
9100	--	--	--	--	--	--	--	Saturated Hydraulic Conductivity, Saturated Leachate Conductivity, and Intrinsic Permeability
9131	--	--	--	--	--	--	--	Total Coliform: Multiple Tube Fermentation Technique
9132	--	--	--	--	--	--	--	Total Coliform: Membrane-Filter Technique

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Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
9200	--	--	Deleted from SW-846 (Up. III)	--	--	--	--	Nitrate
--	--	--	9210 (Up. III)	--	--	9210A	--	Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode
--	--	--	9211 (Up. III)	--	--	--	--	Potentiometric Determination of Bromide in Aqueous Samples with Ion-Selective Electrode
--	--	--	9212 (Up. III)	--	--	--	--	Potentiometric Determination of Chloride in Aqueous Samples with Ion-Selective Electrode
--	--	--	9213 (Up. III)	--	--	--	--	Potentiometric Determination of Cyanide in Aqueous Samples and Distillates with Ion- Selective Electrode
--	--	--	9214 (Up. III)	--	--	--	--	Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Selective Electrode
--	--	--	9215 (Up. III)	--	--	--	--	Potentiometric Determination of Sulfide in Aqueous Samples and Distillates with Ion- Selective Electrode
--	--	--	--	--	9216	--	--	Potentiometric Determination of Nitrite in Aqueous Samples with Ion-Selective Electrode
9250	--	--	--	--	--	--	--	Chloride (Colorimetric, Automated Ferrocyanide AAI)

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Note: The date in parenthesis is the date found at the bottom right-hand corner of the method.

METHOD NUMBER								METHOD TITLE
THIRD ED (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98)	FIN. UP. IIIB (11/04)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER METHODS (e.g., at web site)	
9251	—	—	—	—	—	—	—	Chloride (Colorimetric, Automated Ferricyanide AAI)
9252	—	9252A (Up. II)	Deleted from SW-846 (Up. III)	—	—	—	—	Chloride (Titrimetric, Mercuric Nitrate)
—	—	9253 (Up. II)	—	—	—	—	—	Chloride (Titrimetric, Silver Nitrate)
9310	—	—	—	—	—	—	—	Gross Alpha and Gross Beta
9315	—	—	—	—	—	—	—	Alpha-Emitting Radium Isotopes
9320	—	—	—	—	—	—	—	Radium-228
HCN Test Meth	HCN Test Meth.	HCN Test Method (Up. II)	HCN Test Method (Up. III)	Proposed Removal from SW-846	—	—	—	Test Method to Determine Hydrogen Cyanide Released from Wastes
H ₂ S Test Meth.	H ₂ S Test Meth.	H ₂ S Test Method (Up. II)	H ₂ S Test Method (Up. III)	Proposed Removal from SW-846	—	—	—	Test Method to Determine Hydrogen Sulfide Released from Wastes

STATUS TABLE FOR SW-846 CHAPTER TEXT AND OTHER DOCUMENTS

Note: The date in parenthesis is the date found at the bottom right hand corner of the document.

TITLE	THIRD ED. (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FINAL UP. III (12/96) IIIA 4/98)	DRAFT UP. IVA	DRAFT UP. IVB	FINAL UP. IIIB	CURRENT FINAL VERSION
Disclaimer	–	✓	–	✓ (Up III)	–	–	–	Rev 1 (12/96)
Abstract	✓	✓	✓ (Up. II)	–	–	–	–	Rev 2 (9/94)
Table of Contents	✓	✓	✓ (Up. II & IIB)	✓ (Up. III & IIIA)	✓	✓ (Replaces IVA version)	✓	Rev 6 (11/04)
Method Index and Conversion Table	✓	–	–	–	–	–	–	Rev 0 (9/86)
Preface and Overview	✓	–	–	✓ (Up III)	–	–	–	Rev 1 (12/96)
Acknowledgments	✓	–	–	–	–	–	–	Rev 0 (9/86)
Chapter One – Quality Control	✓	✓	–	–	–		–	Rev 1 (7/92)
Chapter Two – Choosing the Correct Procedure	✓	✓	✓ (Up II)	✓ (Up. III)	✓	✓ (Replaces IVA version)	–	Rev 3 (12/96)
Chapter Three – Inorganic Analytes	✓	✓	✓ (Up II)	✓ (Up III)	✓	✓ (Replaces IVA version)	–	Rev 3 (12/96)

TITLE	THIRD ED. (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FINAL UP. III (12/96) IIIA 4/98)	DRAFT UP. IVA	DRAFT UP. IVB	FINAL UP. IIIB	CURRENT FINAL VERSION
Chapter Four – Organic Analytes	✓	–	✓ (Up. II)	✓ (Up. III)	✓	✓ (Replaces IVA version)	–	Rev 3 (12/96)
Chapter Five – Miscellaneous Test Methods	✓	–	✓ (Up. II)	✓ (Up. III & IIIA)	✓	✓ (Replaces IVA version)	✓	Rev 4 (11/04)
Chapter Six – Properties	✓	–	✓ (Up. II & IIB)	✓ (Up. III)	–	✓	✓	Rev 4 (11/04)
Chapter Seven – Characteristics Introduction and Regulatory Definitions	✓	✓	✓ (Up. II)	✓ (Up. III)	–	–	✓	Rev 4 (11/04)
Chapter Eight –Methods for Determining Characteristics	✓	–	✓ (Up. II)	✓ (Up. III)	–	–	✓	Rev 3 (11/04)
Chapter Nine – Sampling Plan	✓	–	–	–	–	–	–	Rev 0 (9/86)
Chapter Ten – Sampling Methods	✓	–	–	✓ (Up. III)	–	✓	–	Rev 2 (12/96)
Chapter Eleven – Ground Water Monitoring	✓	–	–	–	–	–	–	Rev 0 (9/86)
Chapter Twelve – Land Treatment Monitoring	✓	–	–	–	–	–	–	Rev 0 (9/86)
Chapter Thirteen – Incineration	✓	–	–	–	–	–	–	Rev 0 (9/86)

TITLE	THIRD ED. (9/86)	FINAL UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FINAL UP. III (12/96) IIIA 4/98)	DRAFT UP. IVA	DRAFT UP. IVB	FINAL UP. IIIB	CURRENT FINAL VERSION
Appendix -- Company References	✓	—	—	—	—	—	—	Rev 0 (9/86)

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Method 7380:	Iron (Atomic Absorption, Direct Aspiration)
Method 7381:	Iron (Atomic Absorption, Furnace Technique)
Method 7420:	Lead (Atomic Absorption, Direct Aspiration)
Method 7421:	Lead (Atomic Absorption, Furnace Technique)
Method 7430:	Lithium (Atomic Absorption, Direct Aspiration)
Method 7450:	Magnesium (Atomic Absorption, Direct Aspiration)
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Method 7461:	Manganese (Atomic Absorption, Furnace Technique)
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APPENDIX – COMPANY REFERENCES

NOTE: A suffix of "A" in the method number indicates revision one (the method has been revised once). A suffix of "B" in the method number indicates revision two (the method has been revised twice). A suffix of "C" in the method number indicates revision three (the method has been revised three times), etc. **In order to properly document the method used for analysis, the entire method number including the suffix letter designation (e.g., A, B, C or D) must be identified by the analyst.** A method reference found within the RCRA regulations and the text of SW-846 methods and chapters refers to the latest final revision of the method, even if the method number does not include the appropriate letter suffix.

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4.2.1 Extractions and Preparations

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Method 8131:	Aniline and Selected Derivatives by Gas Chromatography
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Method 8280A:	The Analysis of Polychlorinated Dibenzo- <i>p</i> -Dioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)
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Attachment A: Procedures for the Collection, Handling, Analysis, and Reporting of Wipe Tests Performed within the Laboratory

4.3.3 High Performance Liquid Chromatographic Methods

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CHAPTER FIVE

MISCELLANEOUS TEST METHODS

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. Procedures for required method-defined parameters are not subject to the same flexibility afforded in other SW-846 methods. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

The following methods are found in Chapter Five:

Method 5050:	Bomb Preparation Method for Solid Waste
Method 9010C:	Total and Amenable Cyanide: Distillation
Method 9012B:	Total and Amenable Cyanide (Automated Colorimetric, with Off-Line Distillation)
Method 9013:	Cyanide Extraction Procedure for Solids and Oils
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METHOD 9010C

TOTAL AND AMENABLE CYANIDE DISTILLATION

1.0 SCOPE AND APPLICATION

1.1 This method is a reflux-distillation procedure used to extract soluble cyanide salts and many insoluble cyanide complexes from wastes and leachates. It is based on the decomposition of nearly all cyanides by a reflux distillation procedure using a strong acid and a magnesium catalyst. Cyanide, in the form of hydrocyanic acid (HCN) is purged from the sample and captured into an alkaline scrubber solution. The concentration of cyanide in the scrubber solution is then determined by Method 9014 or Method 9213. This method may be used as a reflux-distillation procedure for both total cyanide and cyanide amenable to chlorination. The "reactive" cyanide content of a waste is not determined by this method. Refer to 40 CFR 261.23 for information on the characteristic of reactivity.

1.2 This method was designed to address the problem of "trace" analyses (<1000 ppm). The method may also be used for "minor" (1000 ppm - 10,000 ppm) and "major" (>10,000 ppm) analyses by adapting the appropriate sample dilution. However, the amount of sodium hydroxide in the standards and the sample analyzed must be the same.

2.0 SUMMARY OF METHOD

2.1 The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide concentration in the absorbing solution is then determined colorimetrically or titrimetrically by Method 9014 or by ion-selective electrode by Method 9213.

3.0 INTERFERENCES

3.1 Interferences are eliminated or reduced by using the distillation procedure. Chlorine and sulfide are interferences in Method 9010.

3.2 Oxidizing agents such as chlorine decompose most cyanides. Chlorine interferences can be removed by adding an excess of sodium arsenite to the waste prior to preservation and storage of the sample to reduce the chlorine to chloride which does not interfere.

3.3 Sulfide interference can be removed by adding an excess of bismuth nitrate to the waste (to precipitate the sulfide) before distillation. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation should be treated by the addition of bismuth nitrate.

3.4 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. The possibility of interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation. Nitrate and nitrite are interferences when present at levels higher than 10 mg/L and in conjunction with certain organic compounds.

3.5 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/L were not found to interfere

3.6 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in high concentrations and may make the endpoint for the titrimetric determination difficult to detect. Refer to Sec. 6.7 for an extraction procedure to eliminate this interference.

4.0 APPARATUS AND MATERIALS

4.1 Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of one liter size with inlet tube and provision for condenser. The gas scrubber may be a 270-mL Fisher-Milligan scrubber (Fisher, Part No. 07-513) or equivalent. The reflux apparatus may be a Wheaton 377160 distillation unit or equivalent.

4.2 Hot plate stirrer/heating mantle

4.3 pH meter.

4.4 Amber light.

4.5 Vacuum source.

4.6 Refrigerator

4.7 Erlenmeyer flask - 500 mL.

4.8 KI starch paper.

4.9 Class A volumetric flasks - 1000, 250, and 100 mL.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Reagents for sample collection, preservation, and handling

5.3.1 Sodium arsenite (0.1N), NaAsO_2 . Dissolve 3.2 g of NaAsO_2 in 250 mL water

5.3.2 Ascorbic acid, $\text{C}_6\text{H}_8\text{O}_6$.

5.3.3 Sodium hydroxide solution (50%), NaOH . Commercially available.

5.3.4 Acetic acid (1.6M) CH_3COOH . Dilute one part of concentrated acetic acid with 9 parts of water

5.3.5 2,2,4-Trimethylpentane, C_8H_{18} .

5.3.6 Hexane, C_6H_{14}

5.3.7 Chloroform, CHCl_3 .

5.4 Reagents for cyanides amenable to chlorination

5.4.1 Calcium hypochlorite solution (0.35M), $\text{Ca}(\text{OCl})_2$. Combine 5 g of calcium hypochlorite and 100 mL of water. Shake before using.

5.4.2 Sodium hydroxide solution (1.25N), NaOH . Dissolve 50 g of NaOH in 1 liter of water

5.4.3 Sodium arsenite (0.1N). See Sec. 5.3.1.

5.4.4 Potassium iodide starch paper.

5.5 Reagents for distillation

5.5.1 Sodium hydroxide (1.25N). See Sec 5.4.2

5.5.2 Bismuth nitrate (0.062M), $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$. Dissolve 30 g of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ in 100 mL of water. While stirring, add 250 mL of glacial acetic acid, CH_3COOH . Stir until dissolved and dilute to 1 liter with water.

5.5.3 Sulfamic acid (0.4N), $\text{H}_2\text{NSO}_3\text{H}$. Dissolve 40 g of $\text{H}_2\text{NSO}_3\text{H}$ in 1 liter of water.

5.5.4 Sulfuric acid (18N), H_2SO_4 . Slowly and carefully add 500 mL of concentrated H_2SO_4 to 500 mL of water.

5.5.5 Magnesium chloride solution (2.5M), $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. Dissolve 510 g of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ in 1 liter of water.

5.5.6 Lead acetate paper.

5.5.7 Stock potassium cyanide solutions -- Refer to Method 9014 for the preparation of stock cyanide solutions and calibration standards.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Samples should be collected in plastic or glass containers. All containers must be thoroughly cleaned and rinsed.

6.2 Oxidizing agents such as chlorine decompose most cyanides. To determine whether oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper. A blue color indicates the need for treatment. Add 0.1N sodium arsenite solution a few mL at a time until a drop of sample produces no color on the indicator paper. Add an

additional 5 mL of sodium arsenite solution for each liter of sample. Ascorbic acid can be used as an alternative although it is not as effective as arsenite. Add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

6.3 Aqueous samples must be preserved by adding 50% sodium hydroxide until the pH is greater than or equal to 12 at the time of collection.

6.4 Samples should be chilled to 4 °C.

6.5 When properly preserved, cyanide samples can be stored for up to 14 days prior to sample preparation steps.

6.6 Solid and oily wastes may be extracted prior to analysis by Method 9013. It uses a dilute NaOH solution (pH = 12) as the extractant. This yields extractable cyanide.

6.7 If fatty acids, detergents, and surfactants are a problem, they may be extracted using the following procedure. Acidify the sample with acetic acid (1.6M) to pH 6.0 to 7.0.

CAUTION: This procedure can produce lethal HCN gas.

Extract with isooctane, hexane, or chloroform (preference in order named) with solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the compounds below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with 50% NaOH solution.

7.0 PROCEDURE

7.1 Pretreatment for cyanides amenable to chlorination

7.1.1 This test must be performed under amber light. $K_3[Fe-(CN)_6]$ may decompose under UV light and hence will test positive for cyanide amenable to chlorination if exposed to fluorescent lighting or sunlight. Two identical sample aliquots are required to determine cyanides amenable to chlorination.

7.1.2 To one 500 mL sample or to a sample diluted to 500 mL, add calcium hypochlorite solution dropwise while agitating and maintaining the pH between 11 and 12 with 1.25N sodium hydroxide until an excess of chlorine is present as indicated by KI-starch paper turning blue. The sample will be subjected to alkaline chlorination by this step.

CAUTION: The initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride; therefore, it is necessary that this reaction be performed in a hood.

7.1.3 Test for excess chlorine with KI-starch paper and maintain this excess for one hour with continuous agitation. A distinct blue color on the test paper indicates a sufficient chlorine level. If necessary, add additional calcium hypochlorite solution.

7.1.4 After one hour, add 1 mL portions of 0.1N sodium arsenite until KI-starch paper shows no residual chlorine. Add 5 mL of excess sodium arsenite to ensure the presence of excess reducing agent.

7.1.5 Analyze the total cyanide concentration of both the chlorinated and the unchlorinated samples by Method 9014 or 9213. The difference between the total cyanide concentration in the chlorinated and unchlorinated samples is equal to the cyanide amenable to chlorination.

7.2 Distillation procedure

7.2.1 Place 500 mL of sample, or sample diluted to 500 mL in the one liter boiling flask. Pipet 50 mL of 1.25N sodium hydroxide into the gas scrubber. If the apparatus in Figure 1 is used, add water until the spiral is covered. Connect the boiling flask, condenser, gas scrubber and vacuum trap.

7.2.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately two bubbles of air per second enter the boiling flask through the air inlet tube.

7.2.3 If samples are known or suspected to contain sulfide, add 50 mL of 0.062M bismuth nitrate solution through the air inlet tube. Mix for three minutes. Use lead acetate paper to check the sample for the presence of sulfide. A positive test is indicated by a black color on the paper.

7.2.4 If samples are known or suspected to contain nitrate or nitrite, or if bismuth nitrate was added to the sample, add 50 mL of 0.4N sulfamic acid solution through the air inlet tube. Mix for three minutes.

NOTE: Excessive use of sulfamic acid could create method bias.

7.2.5 Slowly add 50 mL of 18N sulfuric acid through the air inlet tube. Rinse the tube with water and allow the airflow to mix the flask contents for three minutes. Add 20 mL of 2.5M magnesium chloride through the air inlet and wash the inlet tube with a stream of water.

7.2.6 Heat the solution to boiling. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, and closing the vacuum source, disconnect the gas scrubber.

7.2.7 Transfer the solution from the scrubber into a 250-mL volumetric flask. Rinse the scrubber into the volumetric flask. Dilute to volume with water.

7.2.8 Proceed to the cyanide determinative methods given in Methods 9014 or 9213. If the distillates are not analyzed immediately, they should be stored at 4 °C in tightly sealed flasks.

8.0 QUALITY CONTROL

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

8.2 Employ a minimum of one reagent blank per analytical batch or one in every 20 samples to determine if contamination or any memory effects are occurring

8.3 Analyze check standards with every analytical batch of samples. If the standards are not within 15% of the expected value, then the samples must be reanalyzed

8.4 Run one replicate sample for every 20 samples. A replicate sample is a sample brought through the entire sample preparation and analytical process. The CV of the replicates should be 20% or less. If this criterion is not met, the samples should be reanalyzed

8.5 Run one matrix spiked sample every 20 samples to check the efficiency of sample distillation by adding cyanide from the working standard or intermediate standard to 500 mL of sample to ensure a concentration of approximately 40 µg/L. The matrix spiked sample is brought through the entire sample preparation and analytical process

8.6 It is recommended that at least two standards (a high and a low) be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If distilled standards do not agree within $\pm 10\%$ of the undistilled standards, the analyst should find the cause of the apparent error before proceeding.

8.7 The method of standard additions shall be used for the analysis of all samples that suffer from matrix interferences such as samples which contain sulfides

9.0 METHOD PERFORMANCE

9.1 The titration procedure using silver nitrate is used for measuring concentrations of cyanide exceeding 0.1 mg/L. The colorimetric procedure is used for concentrations below 1 mg/L of cyanide and is sensitive to about 0.02 mg/L.

9.2 EPA Method 335.2 (sample distillation with titration) reports that in a single laboratory using mixed industrial and domestic waste samples at concentrations of 0.06 to 0.62 mg/L CN^- , the standard deviations for precision were ± 0.005 to ± 0.094 , respectively. In a single laboratory using mixed industrial and domestic waste samples at concentrations of 0.28 and 0.62 mg/L CN^- , recoveries (accuracy) were 85% and 102%, respectively.

9.3 In two additional studies using surface water, ground water, and landfill leachate samples, the titration procedure was further evaluated. The concentration range used in these studies was 0.5 to 10 mg/L cyanide. The detection limit was found to be 0.2 mg/L for both total and amenable cyanide determinations. The precision (CV) was 6.9 and 2.6 for total cyanide determinations and 18.6 and 9.1 for amenable cyanide determinations. The mean recoveries were 94% and 98.9% for total cyanide, and 86.7% and 97.4% for amenable cyanide.

10.0 REFERENCES

1. 1985 Annual Book of ASTM Standards, Vol. 11.01, "Standard Specification for Reagent Water"; ASTM. Philadelphia, PA, 1985., D1193-77.
2. 1982 Annual Book of ASTM Standards, Part 19, "Standard Test Methods for Cyanide in Water"; ASTM: Philadelphia, PA, 1982; 2036-82.
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FIGURE 1.
APPARATUS FOR CYANIDE DISTILLATION

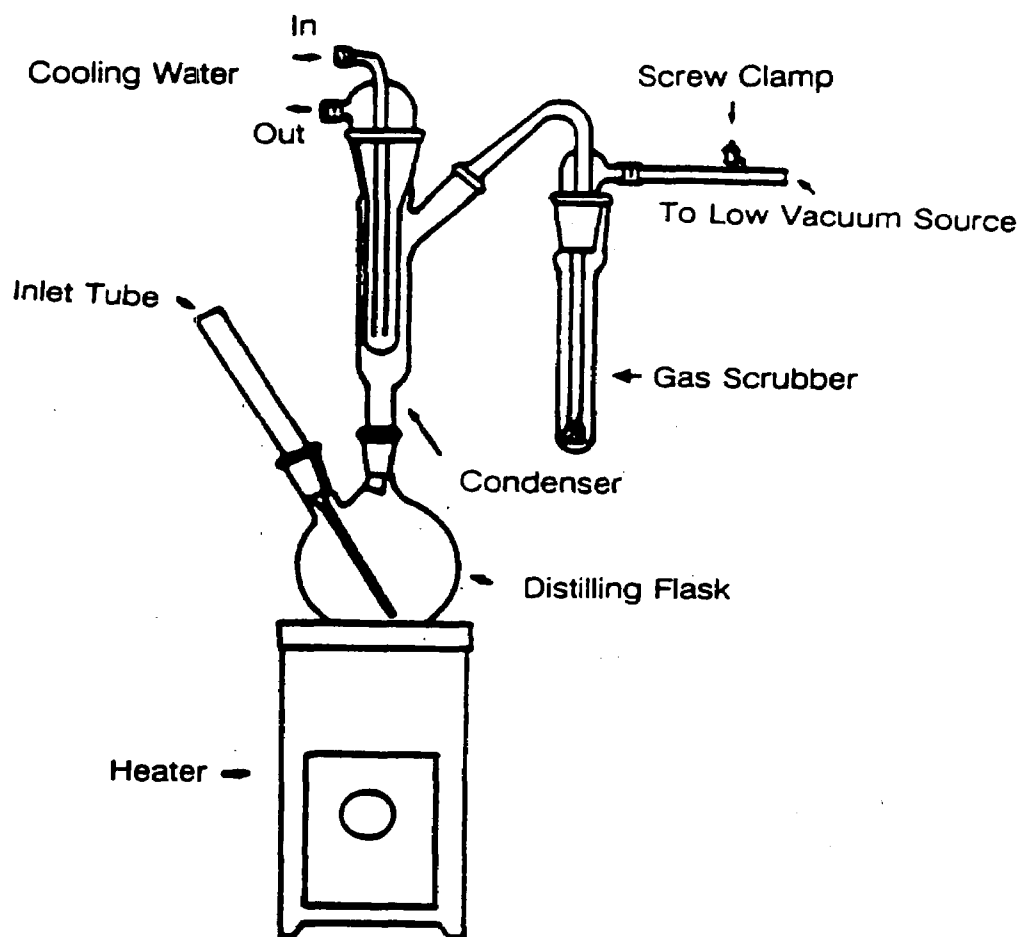
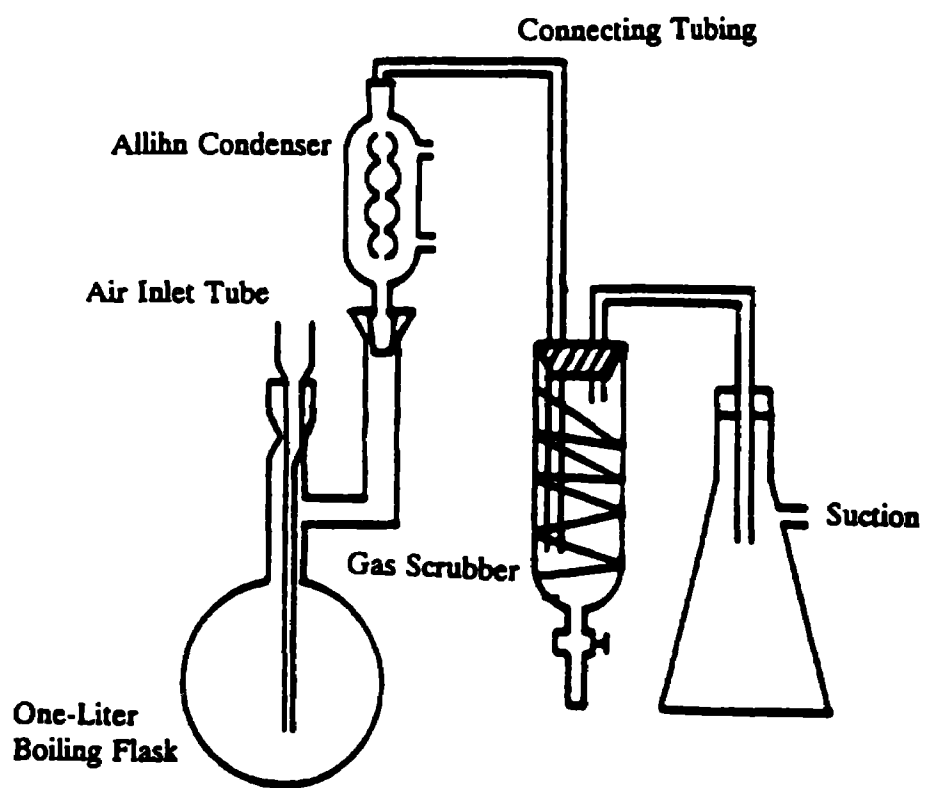
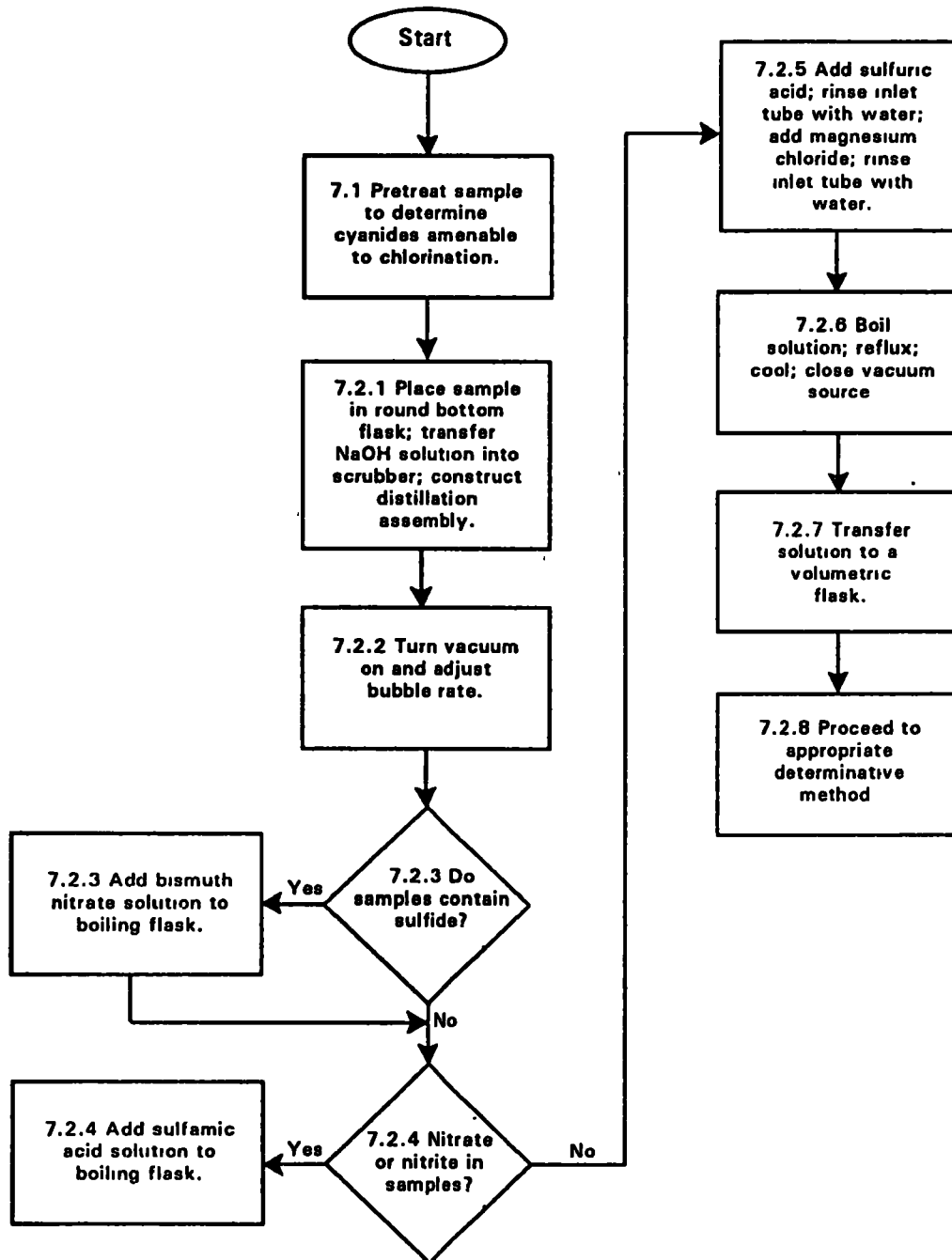


FIGURE 2.
APPARATUS FOR CYANIDE DISTILLATION



METHOD 9010C

TOTAL AND AMENABLE CYANIDE: DISTILLATION



METHOD 9012B

TOTAL AND AMENABLE CYANIDE (AUTOMATED COLORIMETRIC, WITH OFF-LINE DISTILLATION)

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of inorganic cyanide (CAS Registry Number 57-12-5) in wastes or leachate. This method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide and cyanide amenable to chlorination. The "reactive" cyanide content of a waste is not determined by this method. Refer to 40 CFR 261.23 for information on the characteristic of reactivity.

2.0 SUMMARY OF METHOD

2.1 The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated UV colorimetry.

2.2 In the automated colorimetric measurement, the cyanide is converted to cyanogen chloride (CNCl) by reaction with Chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The concentration of NaOH must be the same in the standards, the scrubber solutions, and any dilution of the original scrubber solution to obtain colors of comparable intensity.

3.0 INTERFERENCES

3.1 Interferences are eliminated or reduced by using the distillation procedure. Chlorine and sulfide are interferences in this method.

3.2 Oxidizing agents such as chlorine decompose most cyanides. Chlorine interferences can be removed by adding an excess of sodium arsenite to the waste prior to preservation and storage of the sample to reduce the chlorine to chloride which does not interfere.

3.3 Sulfide interference can be removed by adding an excess of bismuth nitrate to the waste (to precipitate the sulfide) before distillation. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation should be treated by the addition of bismuth nitrate.

3.4 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. The possibility of interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation. Nitrate and nitrite are interferences when present at levels higher than 10 mg/L and in conjunction with certain organic compounds.

3.5 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/L were not found to interfere in Method 9010.

3.6 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in large concentrations and will make the endpoint of the titration difficult to detect. They may be extracted at pH 6-7.

4.0 APPARATUS AND MATERIALS

4.1 Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of one liter size with inlet tube and provision for condenser. The gas scrubber may be a 270-mL Fisher-Milligan scrubber (Fisher, Part No. 07-513 or equivalent). The reflux apparatus may be a Wheaton 377160 distillation unit or equivalent

4.2 Automated continuous-flow analytical instrument with:

4.2.1 Sampler.

4.2.2 Manifold.

4.2.3 Proportioning pump.

4.2.4 Heating bath with distillation coil

4.2.5 Distillation head.

4.2.6 Colorimeter equipped with a 15-mm flowcell and 570 nm filter.

4.2.7 Recorder.

4.3 Hot plate stirrer/heating mantle.

4.4 pH meter.

4.5 Amber light.

4.6 Vacuum source

4.7 Refrigerator.

4.8 5 mL microburette.

4.9 7 Class A volumetric flasks -- 100 and 250 mL.

4.10 Erlenmeyer flask -- 500 mL.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other

grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination

5.2 Reagent water All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Reagents for sample collection, preservation, and handling

5.3.1 Sodium arsenite (0.1N), NaAsO_2 . Dissolve 3.2 g of NaAsO_2 in 250 mL water.

5.3.2 Ascorbic acid, $\text{C}_6\text{H}_8\text{O}_6$.

5.3.3 Sodium hydroxide solution (50%), NaOH . Commercially available.

5.3.4 Acetic acid (1.6M) CH_3COOH . Dilute one part of concentrated acetic acid with 9 parts of water.

5.3.5 2,2,4-Trimethylpentane, C_8H_{18} .

5.3.6 Hexane, C_6H_{14} .

5.3.7 Chloroform, CHCl_3 .

5.4 Reagents for cyanides amenable to chlorination

5.4.1 Calcium hypochlorite solution (0.35M), $\text{Ca}(\text{OCl})_2$. Combine 5 g of calcium hypochlorite and 100 mL of water. Shake before using.

5.4.2 Sodium hydroxide solution (1.25N), NaOH . Dissolve 50 g of NaOH in 1 liter of water.

5.4.3 Sodium arsenite (0.1N). See Sec 5.3.1.

5.4.4 Potassium iodide starch paper.

5.5 Reagents for distillation

5.5.1 Sodium hydroxide (1.25N). See Sec. 5.4.2.

5.5.2 Bismuth nitrate (0.062M), $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$. Dissolve 30 g of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ in 100 mL of water. While stirring, add 250 mL of glacial acetic acid, CH_3COOH . Stir until dissolved and dilute to 1 liter with water.

5.5.3 Sulfamic acid (0.4N), $\text{H}_2\text{NSO}_3\text{H}$. Dissolve 40 g $\text{H}_2\text{NSO}_3\text{H}$ in 1 liter of water.

5.5.4 Sulfuric acid (18N), H_2SO_4 . Slowly and carefully add 500 mL of concentrated H_2SO_4 to 500 mL of water.

5.5.5 Magnesium chloride solution (2.5M), $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. Dissolve 510 g of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ in 1 liter of water.

5 5 6 Lead acetate paper.

5.6 Reagents for automated colorimetric determination

5 6 1 Pyridine-barbituric acid reagent -- Place 15 g of barbituric acid in a 250-mL volumetric flask, add just enough reagent water to wash the sides of the flask, and wet the barbituric acid. Add 75 mL of pyridine and mix. Add 15 mL of concentrated HCl, mix, and cool to room temperature. Dilute to 250 mL with reagent water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.

5.6.2 Chloramine-T solution -- Dissolve 2.0 g of white, water soluble chloramine-T in 500 mL of reagent water and refrigerate until ready to use.

5.6.3 Sodium hydroxide, 1 N -- Dissolve 40 g of NaOH in reagent water, and dilute to 1 liter.

5.6.4 All working standards should contain 2 mL of 1 N NaOH (Sec. 5.6.3) per 100 mL

5.6.5 Dilution water and receptacle wash water (NaOH, 0.25 N) -- Dissolve 10.0 g of NaOH in 500 mL of reagent water. Dilute to 1 liter

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Samples should be collected in plastic or glass containers. All containers must be thoroughly cleaned and rinsed.

6.2 Oxidizing agents such as chlorine decompose most cyanides. To determine whether oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper. A blue color indicates the need for treatment. Add 0.1N sodium arsenite solution a few mL at a time until a drop of sample produces no color on the indicator paper. Add an additional 5 mL of sodium arsenite solution for each liter of sample. Ascorbic acid can be used as an alternative although it is not as effective as arsenite. Add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

6.3 Aqueous samples must be preserved by adding 50% sodium hydroxide until the pH is greater than or equal to 12 at the time of collection.

6.4 Samples should be chilled to 4 °C.

6.5 When properly preserved, cyanide samples can be stored for up to 14 days prior to sample preparation steps.

6.6 Solid and oily wastes may be extracted prior to analysis by Method 9013 (Cyanide Extraction Procedure for Solids and Oils). It uses a dilute NaOH solution (pH = 12) as the extractant. This yields extractable cyanide.

6.7 If fatty acids, detergents, and surfactants are a problem, they may be extracted using the following procedure. Acidify the sample with acetic acid (1.6M) to pH 6.0 to 7.0.

CAUTION: This procedure can produce lethal HCN gas.

Extract with isooctane, hexane, or chloroform (preference in order named) with solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the compounds below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with 50% NaOH solution.

7.0 PROCEDURE

7.1 Pretreatment for cyanides amenable to chlorination

7.1.1 This test must be performed under amber light. $K_3[Fe-(CN)_6]$ may decompose under UV light and hence will test positive for cyanide amenable to chlorination if exposed to fluorescent lighting or sunlight. Two identical sample aliquots are required to determine cyanides amenable to chlorination.

7.1.2 To one 500 mL sample or to a sample diluted to 500 mL, add calcium hypochlorite solution dropwise while agitating and maintaining the pH between 11 and 12 with 1.25N sodium hydroxide until an excess of chlorine is present as indicated by KI-starch paper turning blue. The sample will be subjected to alkaline chlorination by this step.

CAUTION: The initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride; therefore, it is necessary that this reaction be performed in a hood.

7.1.3 Test for excess chlorine with KI-starch paper and maintain this excess for one hour with continuous agitation. A distinct blue color on the test paper indicates a sufficient chlorine level. If necessary, add additional calcium hypochlorite solution.

7.1.4 After one hour, add 1 mL portions of 0.1N sodium arsenite until KI-starch paper shows no residual chlorine. Add 5 mL of excess sodium arsenite to ensure the presence of excess reducing agent.

7.1.5 Test for total cyanide as described below in both the chlorinated and the unchlorinated samples. The difference of total cyanide in the chlorinated and unchlorinated samples is the cyanide amenable to chlorination.

7.1.6 If samples are known or suspected to contain sulfide, add 50 mL of 0.062M bismuth nitrate solution through the air inlet tube. Mix for three minutes. Use lead acetate paper to check the sample for the presence of sulfide. A positive test is indicated by a black color on the paper.

7.2 Distillation procedure

7.2.1 Place 500 mL of sample, or sample diluted to 500 mL in the one liter boiling flask. Pipet 50 mL of 1.25N sodium hydroxide into the gas scrubber. If the apparatus in Figure 1 is used, add water until the spiral is covered. Connect the boiling flask, condenser, gas scrubber and vacuum trap.

7.2.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately two bubbles of air per second enter the boiling flask through the air inlet tube.

7.2.3 If samples are known or suspected to contain nitrate or nitrite, or if bismuth nitrate was added to the sample, add 50 mL of 0.4N sulfamic acid solution through the air inlet tube. Mix for three minutes.

NOTE. Excessive use of sulfamic acid could create method bias.

7.2.4 Slowly add 50 mL of 18N sulfuric acid through the air inlet tube. Rinse the tube with water and allow the airflow to mix the flask contents for three minutes. Add 20 mL of 2.5M magnesium chloride through the air inlet and wash the inlet tube with a stream of water.

7.2.5 Heat the solution to boiling. Reflux for one hour. Turn off heat and continue the airflow for at least 15 min. After cooling the boiling flask, and closing the vacuum source, disconnect the gas scrubber.

7.2.6 Transfer the solution from the scrubber into a 250-mL volumetric flask. Rinse the scrubber into the volumetric flask. Dilute to volume with water.

7.3 Automated colorimetric determination

7.3.1 Set up the manifold in a hood or a well-ventilated area as shown in Figure 3.

7.3.2 Allow colorimeter and recorder to warm up for 30 min. Run a baseline with all reagents, feeding reagent water through the sample line.

7.3.3 Place appropriate standards in the sampler in order of increasing concentration. Complete loading of the sampler tray with unknown samples.

7.3.4 When the baseline becomes steady, begin the analysis

7.4 Standard curve for samples without sulfide

7.4.1 Prepare a series of standards by pipetting suitable volumes of working standard potassium cyanide solution into 250-mL volumetric flasks. To each flask, add 50 mL of 1.25N sodium hydroxide and dilute to 250 mL with water. Prepare using the following table. The sodium hydroxide concentration will be 0.25N.

mL of Working Standard Solution (1 mL = 10 µg CN ⁻)	Concentration (µg CN ⁻ /L)
0.0	Blank
1.0	40
2.0	80
5.0	200
10.0	400
15.0	600
20.0	800

7.4.2 After the standard solutions have been prepared according to the table above, pipet 50 mL of each standard solution into a 100-mL volumetric flask and proceed

to Secs. 7.3.2 and 7.3.3 to obtain absorbance values for the standard curve. The final concentrations for the standard curve will be one half of the amounts in the above table (final concentrations ranging from 20 to 400 µg/L).

7.4.3 It is recommended that at least two standards (a high and a low) be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If distilled standards do not agree within $\pm 10\%$ of the undistilled standards, the analyst should find the cause of the apparent error before proceeding.

7.4.4 Prepare a standard curve ranging from 20 to 400 µg/L by plotting absorbance of standard versus the cyanide concentration.

7.5 Standard curve for samples with sulfide

7.5.1 It is imperative that all standards be distilled in the same manner as the samples using the method of standard additions (for example, bismuth nitrate must also be added to the standards). Standards distilled by this method will give a linear curve, at low concentrations, but as the concentration increases, the recovery decreases. It is recommended that at least five standards be distilled.

7.5.2 Prepare a series of standards similar in concentration to those mentioned in Sec. 7.4.1 and analyze as in Sec. 7.3. Prepare a standard curve by plotting absorbance of standard versus the cyanide concentration.

7.6 Calculation -- Prepare a standard curve by plotting peak heights of standards against their concentration values. Compute concentrations of samples by comparing sample peak heights with the standard curve.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures.

8.2 Verify the calibration curve with an independent calibration check standard. If the standards are not within 15% of the expected value, a new recalibration curve is required. Verify the calibration curve with every sample batch by analyzing a mid-range standard.

8.3 Run one matrix spike sample for every 10 samples to check the efficiency of sample distillation. A matrix spike should be prepared by adding cyanide from the working standard or intermediate standard to 500 mL of sample to ensure a concentration of approximately 40 µg/L. Both the matrix duplicate and matrix spike duplicate are brought through the entire sample preparation and analytical process.

8.4 The method of standard additions shall be used for the analysis of all samples that suffer from matrix interferences such as samples which contain sulfides.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are not available at this time.

10.0 REFERENCES

- 1. Annual Book of ASTM Standards, Part 31, "Water," Standard D2036-75, Method B, p. 505 (1976).**
- 2. Goulden, P.D., B.K. Afghan, and P. Brooksbank, Determination of Nanogram Quantities of Simple and Complex Cyanides in Water, Anal. Chem., 44(11), pp. 1845-49 (1972).**
- 3. Standard Methods for the Examination of Water and Wastewater, 14th ed., pp. 376 and 370, Method 413F and D (1975).**
- 4. Technicon AutoAnalyzer II Methodology, Industrial Method No. 315-74 WCUV Digestion and Distillation, Technicon Industrial Systems, Tarrytown, New York, 10591 (1974).**

FIGURE 1
APPARATUS FOR CYANIDE DISTILLATION

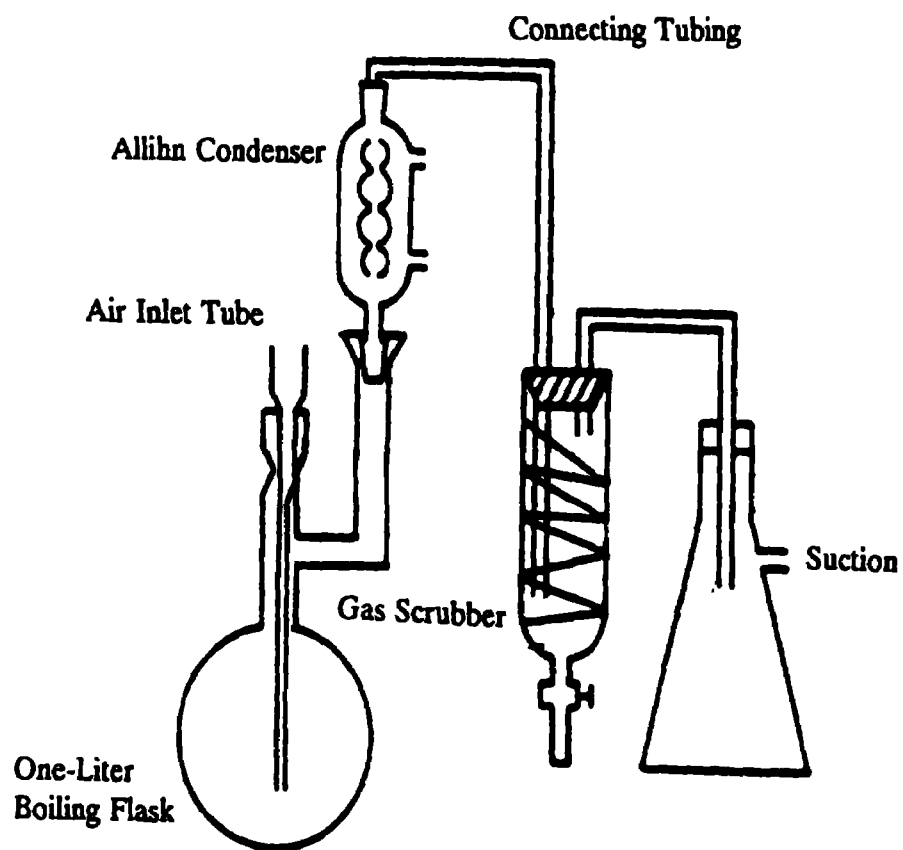


FIGURE 2
CYANIDE DISTILLATION APPARATUS

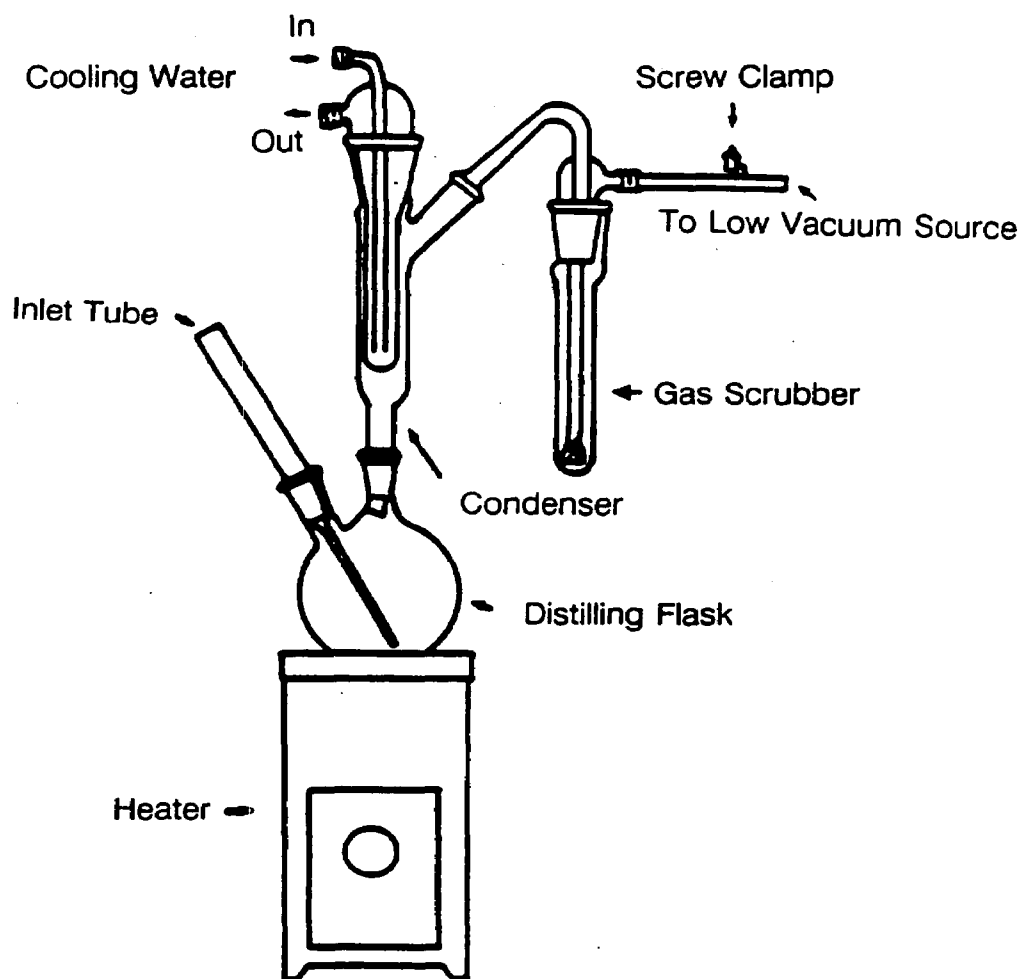
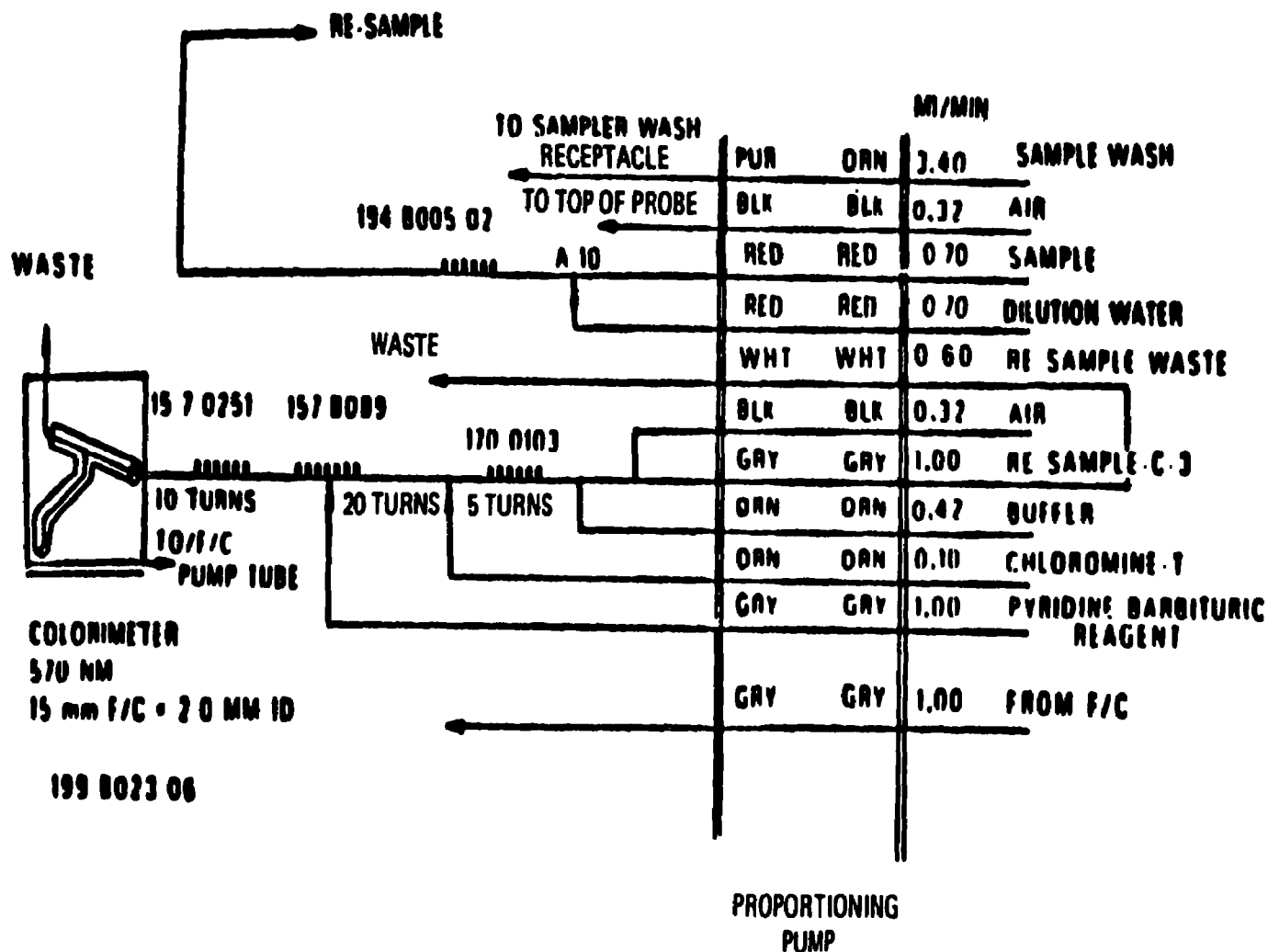
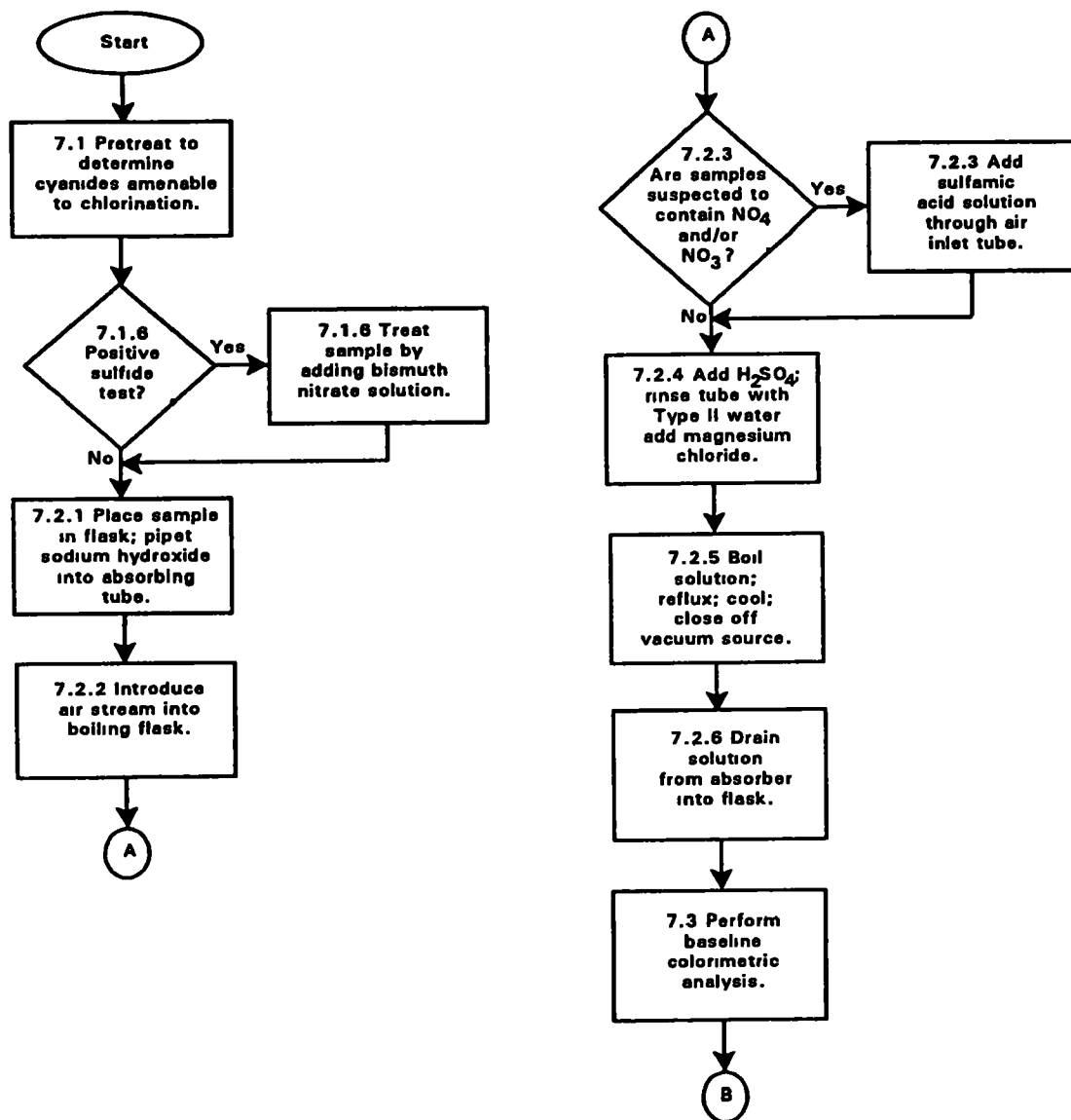


FIGURE 3
CYANIDE MANIFOLD AA11

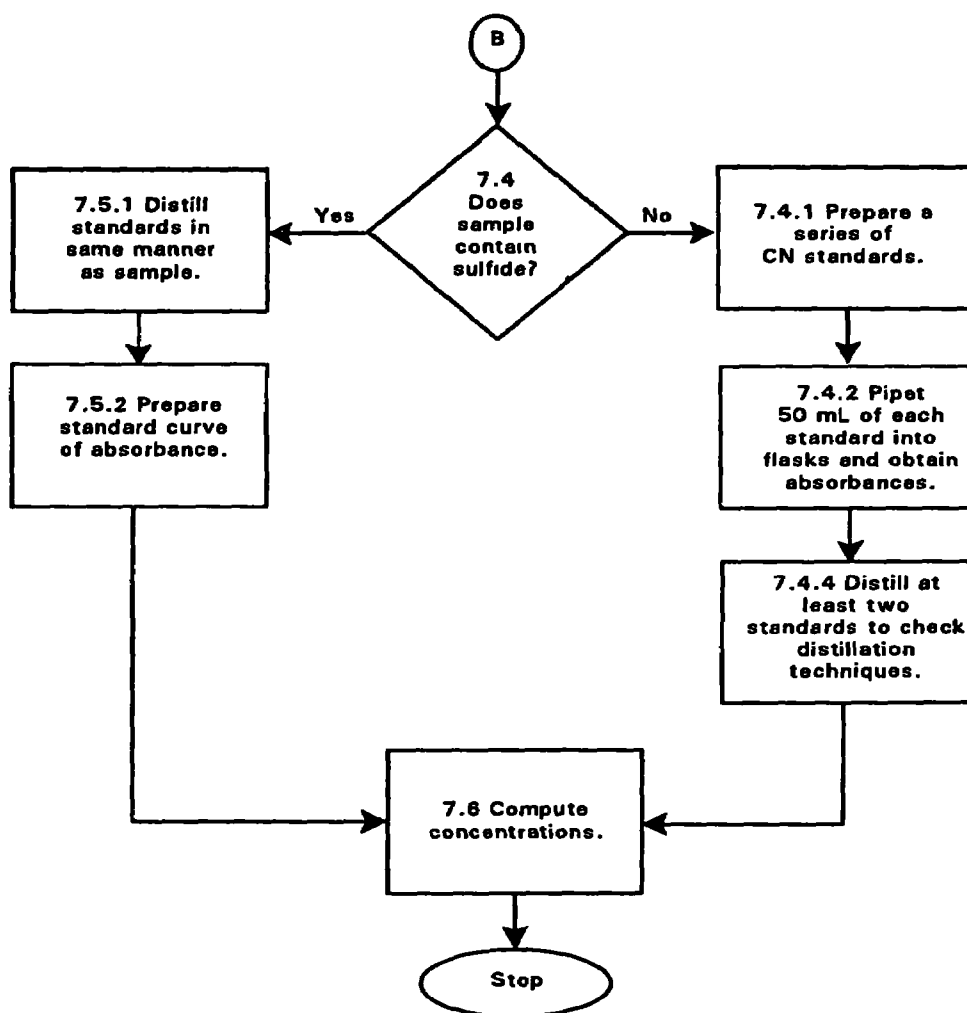


METHOD 9012B

TOTAL AND AMENABLE CYANIDE (AUTOMATED COLORIMETRIC
WITH OFF-LINE DISTILLATION)



METHOD 9012B (continued)



METHOD 9060A

TOTAL ORGANIC CARBON

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of organic carbon in ground water, surface and saline waters, and domestic and industrial wastes. Some restrictions are noted in Secs. 2.0 and 3.0.

1.2 This method is most applicable to measurement of organic carbon above 1 mg/L.

2.0 SUMMARY OF METHOD

2.1 Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide (CO_2) by either catalytic combustion or wet chemical oxidation. The CO_2 formed is then either measured directly by an infrared detector or converted to methane (CH_4) and measured by a flame ionization detector. The amount of CO_2 or CH_4 in a sample is directly proportional to the concentration of carbonaceous material in the sample.

2.2 Carbonaceous analyzers are capable of measuring all forms of carbon in a sample. However, because of various properties of carbon-containing compounds in liquid samples, the manner of preliminary sample treatment as well as the instrument settings will determine which forms of carbon are actually measured. The forms of carbon that can be measured by this method are:

1. Soluble, nonvolatile organic carbon e.g., natural sugars
2. Soluble, volatile organic carbon. e.g., mercaptans, alkanes, low molecular weight alcohols.
3. Insoluble, partially volatile carbon. e.g., low molecular weight oils
4. Insoluble, particulate carbonaceous materials. e.g., cellulose fibers.
5. Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter e.g., oily matter adsorbed on silt particles.

2.3 Carbonate and bicarbonate are inorganic forms of carbon and must be separated from the total organic carbon value. Depending on the instrument manufacturer's instructions, this separation can be accomplished by either a simple mathematical subtraction, or by removing the carbonate and bicarbonate by converting them to CO_2 with degassing prior to analysis.

3.0 INTERFERENCES

3.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.

3.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter-type syringe or pipet. The openings of the syringe or pipet limit the maximum size of particle which may be included in the sample.

3.3 Removal of carbonate and bicarbonate by acidification and purging with nitrogen, or other inert gas, can result in the loss of volatile organic substances.

4.0 APPARATUS AND MATERIALS

4.1 Apparatus for blending or homogenizing samples -- Generally, a Waring-type blender is satisfactory

4.2 Apparatus for total and dissolved organic carbon

4.2.1 Several companies manufacture analyzers for measuring carbonaceous material in liquid samples. The most appropriate system should be selected based on consideration of the types of samples to be analyzed, the expected concentration range, and the forms of carbon to be measured.

4.2.2 No specific analyzer is recommended as superior. If the technique of chemical oxidation is used, the laboratory must be certain that the instrument is capable of achieving good carbon recoveries in samples containing particulates.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193) -- Water should be monitored for impurities, and should be boiled and cooled to remove CO₂

5.2 Potassium hydrogen phthalate, stock solution, 1,000 mg/L carbon -- Dissolve 0.2128 g of potassium hydrogen phthalate (primary standard grade) in Type II water and dilute to 100.0 mL

NOTE: Sodium oxalate and acetic acid are not recommended as stock solutions

5.3 Potassium hydrogen phthalate, standard solutions -- Prepare standard solutions from the stock solution by dilution with Type II water.

5.4 Carbonate-bicarbonate, stock solution, 1,000 mg/L carbon -- Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100-mL volumetric flask. Dissolve with Type II water.

5.5 Carbonate-bicarbonate, standard solution -- Prepare a series of standards similar to Step 5.3.

NOTE: This standard is not required by some instruments.

5.6 Blank solution -- Use the same Type II water as was used to prepare the standard solutions

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.

NOTE: A brief study performed in the EPA Laboratory indicated that Type II water stored in new, 1-qt cubitainers did not show any increase in organic carbon after 2 weeks' exposure.

6.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the time between sample collection and the start of analysis should be minimized. Also, samples should be kept cool (4 °C) and protected from sunlight and atmospheric oxygen.

6.3 In instances where analysis cannot be performed within 2 hr from time of sampling, the sample is acidified ($\text{pH} \leq 2$) with HCl or H_2SO_4 .

7.0 PROCEDURE

7.1 Homogenize the sample in a blender.

NOTE: To avoid erroneously high results, inorganic carbon must be accounted for. The preferred method is to measure total carbon and inorganic carbon and to obtain the organic carbon by subtraction. If this is not possible, follow Steps 7.2 and 7.3 prior to analysis; however, volatile organic carbon may be lost.

7.2 Lower the pH of the sample to 2.

7.3 Purge the sample with nitrogen for 10 min.

7.4 Follow instrument manufacturer's instructions for calibration, procedure, and calculations.

7.5 For calibration of the instrument, a series of standards should be used that encompasses the expected concentration range of the samples.

7.6 Quadruplicate analysis is required. Report both the average and the range.

8.0 QUALITY CONTROL

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

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

8.3 Verify calibration with an independently prepared check standard every 15 samples.

8.4 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process.

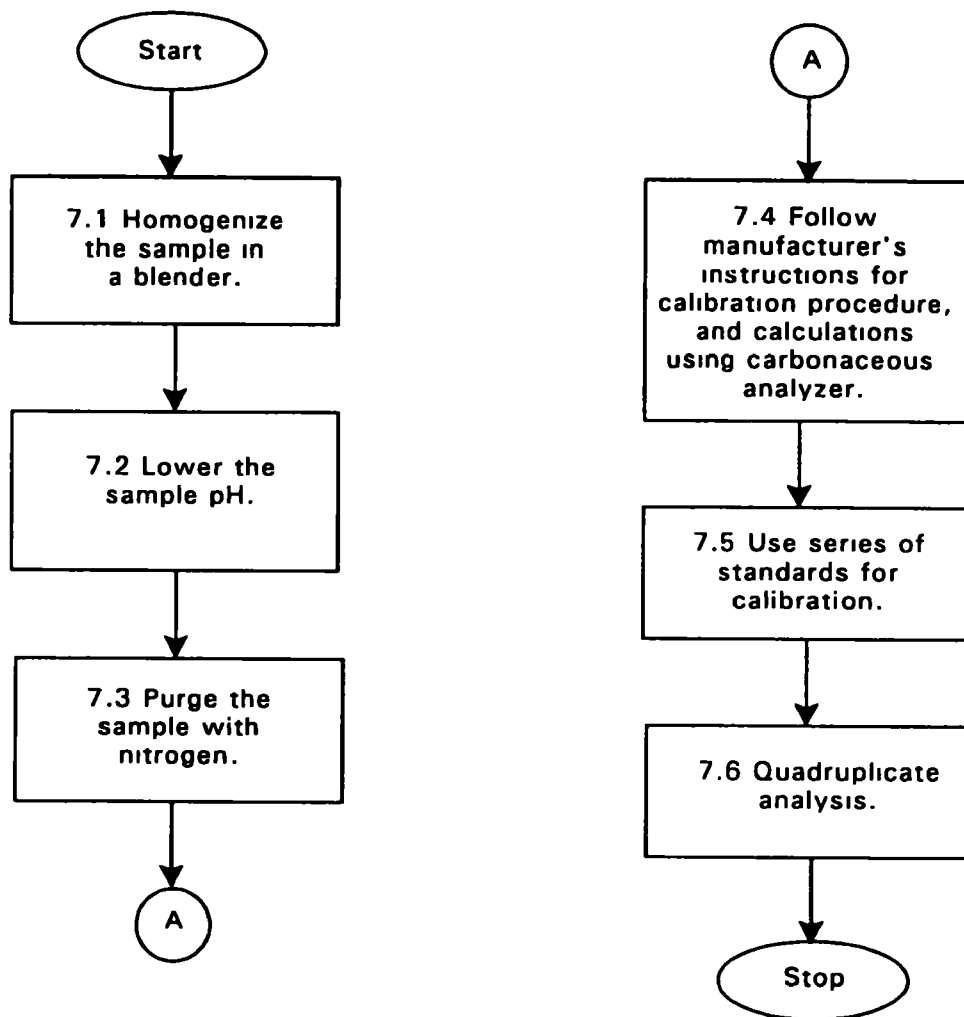
9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 415.1 of Methods for Chemical Analysis of Water and Wastes.

10.0 REFERENCES

- 1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).**
- 2. Standard Methods for the Examination of Water and Wastewater, 14th ed., p. 532, Method 505 (1975).**

**Method 9060A
TOTAL ORGANIC CARBON**



METHOD 9070A

n-HEXANE EXTRACTABLE MATERIAL (HEM) FOR AQUEOUS SAMPLES

See Method 1664, Revision A, Publication No. EPA-821-R-98-002, for this method procedure.

The full text of EPA Method 1664, Revision A, may be viewed or downloaded from the Internet at : <http://www.epa.gov/ost/methods/1664f051.html>.

You may also obtain copies of Method 1664, Revision A, through the U.S. EPA National Service Center for Environmental Publications (NSCEP), 11029 Kenwood Road, Cincinnati, OH 45242.

METHOD 9071B

n-HEXANE EXTRACTABLE MATERIAL (HEM) FOR SLUDGE, SEDIMENT, AND SOLID SAMPLES

1.0 SCOPE AND APPLICATION

1.1 Method 9071 may be used to quantify low concentrations of oil and grease in soil, sediments, sludges, and other solid materials amenable to chemical drying and solvent extraction with n-hexane. "Oil and grease" is a conventional pollutant under 40 CFR 401.16 and generally refers to substances, including biological lipids and mineral hydrocarbons, that have similar physical characteristics and common solubility in an organic extracting solvent. As such, oil and grease is an operationally defined parameter, and the results will depend entirely on the extracting solvent and method of extraction. Method 9071 employs n-hexane as the extraction solvent with Soxhlet extraction and the results of this method are appropriately termed "n-hexane extractable material (HEM)." Section 1.2 lists the type of materials that may be extracted by this method. In the context of this method, "HEM" is used throughout this method and for operational purposes, may be considered synonymous with "oil and grease" within the limitations discussed below.

1.2 Specifically, Method 9071 is suitable for extracting relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, biological lipids, and related materials.

1.3 Method 9071 is not recommended for measuring materials that volatilize at temperatures below 85°C. Petroleum fuels from gasoline through #2 fuel oil may be partially lost during the solvent removal process.

1.4 Some crude oils and heavy fuel oils may contain materials that are not soluble in n-hexane, and recovery of these materials may be low.

2.0 SUMMARY OF METHOD

2.1 A representative portion of wet (as received) waste is acidified with concentrated HCl and chemically dried with magnesium sulfate or sodium sulfate. Magnesium sulfate monohydrate is used to dry acidified sludges as it will combine with 75% of its own weight in water in forming $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. Anhydrous sodium sulfate is used to dry soil and sediment samples.

2.2 After drying, the HEM is extracted with n-hexane using a Soxhlet apparatus. The n-hexane extract is then distilled from the extract and the HEM is desiccated and weighed.

2.3 When necessary, a separate sample portion is evaluated for percent solids, and the dry weight fraction may be used to calculate the dry-weight HEM concentration of the soil, sediment, or waste.

3.0 DEFINITIONS

3.1 n-Hexane extractable material (HEM, oil and grease): Material that is extracted from a sample using n-hexane and determined by this method. This material includes relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related matter.

3.2 Refer to Chapter One for additional definitions.

4.0 INTERFERENCES

4.1 This method is entirely empirical, and duplicate results having a high degree of precision can be obtained only by strict adherence to all details. The rate of cycling and time of extraction in the Soxhlet apparatus must be consistent and length of time required for drying and cooling extracted materials must be the same in order to generate consistent results. It is important that the procedures be performed as directed due to the varying solubilities of the different greases and heavy mineral oils.

4.2 Solvents, reagents, glassware, and other sample-processing hardware may yield artifacts that could affect the results. All solvents and reagents used in the analysis should be demonstrated to be free from interferences by processing a method blank with each analytical batch. Specific selection of reagents, solvent washes, or purification of solvents may be required. Use of plastic measuring devices, and/or plastic tubing attachments must be avoided.

4.3 Glassware should be cleaned by washing with hot tap water with detergent, rinsing with tap water and reagent water, and rinsing with solvent. Glassware may also be baked at 200-250°C for 1 hour. Boiling flasks that are used to contain the extracted residues may be dried in an oven at 105-115°C and stored in a desiccator until used. Depending on the project DQOs, strict adherence to the washing and handling procedures cited above may not be necessary as long as the laboratory can demonstrate that alternative cleaning procedures yield acceptable method performance and meet method blank acceptance criteria.

4.4 A gradual increase in weight may result due to the absorption of oxygen; a gradual loss of weight may result due to volatilization. Extracted residues should be maintained in a desiccator during cooling and prior to weighing. Extracted residues should be weighed as soon as possible after cooling.

4.5 The presence of non-oily extractable substance such as sulfur compounds, organic dyes, and chlorophyll, may result in a positive bias. For the purpose of this method, all materials extracted and retained during this procedure are defined as HEM.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst that uses this method. This monitoring should be performed using Occupational Safety and Health Administration (OSHA) or National Institute of Occupational Safety and Health (NIOSH) approved personal hygiene monitoring methods. Results of this monitoring should be made available to the analyst.

5.2 n-Hexane has been shown to have increased neurotoxic effects over other hexanes and some other solvents. OSHA has proposed a time-weighted average (TWA) of 50 parts-per-million (ppm); NIOSH concurs that an 8-hour TWA/permissible exposure limit (PEL) of 50 ppm is appropriate for n-hexane; and the American Conference of Governmental Industrial Hygienists (ACGIH) has published a threshold limit value (TLV) of 50 ppm for n-hexane. Inhalation of n-hexane should be minimized by performing all operations with n-hexane in a explosion-proof hood or well-ventilated area.

5.3 n-Hexane has a flash point of -23°C (-9°F), has explosive limits in air in the range of 1 to 7 percent, and poses a serious fire risk when heated or exposed to flame. n-Hexane can react vigorously with oxidizing materials. The laboratory should include procedures in its operations that address the safe handling of n-hexane.

5.4 Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

5.5 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

6.1 Soxhlet extraction apparatus.

6.2 Heating mantle - explosion-proof, with temperature control.

6.3 Boiling flask - 125-mL or appropriate size.

6.4 Analytical balance - capable of weighing 0.1 mg.

6.5 Vacuum pump, or other vacuum source.

6.6 Paper extraction thimble for Soxhlet apparatus.

6.7 Glass wool or small glass beads to fill thimble.

6.8 Grease-free, non-absorbent cotton - To remove possible interferences, each batch of cotton should be washed with n-hexane. Solvent washing may not be necessary if the laboratory can demonstrate that the unwashed cotton does not affect the performance of the method or that the concentration of HEM in the sample is so high that low contaminant concentration is insignificant.

6.9 Beakers - 100- 150-mL.

6.10 pH paper.

6.11 Porcelain mortar and pestle.

6.12 Extraction flask - 150-mL or appropriate size.

6.13 Waterbath or steam bath-explosion-proof - capable of maintaining a temperature of at least 85°C.

6.14 Distilling apparatus - For removing n-hexane from extract.

6.14.1 Distilling head-Claisen (VWR Scientific No 26339-005, or equivalent), includes Claisen-type connecting tube and condenser.

6.14.2 Distillation adapter (used to attach distilling head and to the waste collection flask for recovery of solvent).

6.14.3 Distillate collection flask (attached to the distilling adaptor for collection of the distilled solvent).

6.14.4 Ice bath or recirculating chiller (to aid in the condensation and collection of the distilled solvent).

6.15 Desiccator - Cabinet or jar type, capable of holding boiling flasks during cooling and storage.

6.16 Tongs - for handling the boiling flasks.

6.17 Glass fiber filter paper - Whatman No. 40 or equivalent.

6.18 Boiling chips - Silicon carbide or fluoropolymer.

7.0 REAGENTS

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

7.3 Concentrated hydrochloric acid (HCl).

7.4 Magnesium sulfate monohydrate. Prepare $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ by spreading a thin layer in a dish and drying in an oven at 150°C overnight. Store in a tightly sealed glass container until used.

7.5 Sodium sulfate, granular, anhydrous (Na_2SO_4). Purify by heating at 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate. Store in a tightly sealed glass container until used.

7.6 n-Hexane. Purity of 85%, 99.0% minimum saturated C_6 isomers, residue less than 1 mg/L. Boiling point, 69°C .

7.7 Hexadecane($\text{CH}_3(\text{CH}_2)_{14}\text{CH}_3$)/stearic acid ($\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$). 1:1 spiking solution. Prepare in acetone at a concentration of 2 mg/mL each.

Weigh 200 ± 2 mg of stearic acid and 200 ± 2 mg hexadecane into a 100 mL volumetric flask and fill to the mark with acetone. The total concentration of this stock is 4000 mg/L (ppm) HEM. This standard may be used for spiking samples and preparing laboratory control samples. Store in a glass container with a fluoropolymer-lined cap at room temperature. Shield from light.

Note: The spiking solution may require warming for complete dissolution of stearic acid.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 A minimum of 100 grams of sample should be collected using a metal spatula, spoon, or equivalent device. Samples should be collected into a pre-cleaned wide-mouth glass container fitted with a TFE-lined screw cap.

8.2 When practical (i.e., when the sample matrix allows the complete mixing of sample and acid such as with a pourable sludge or sediment), the sample should be preserved to a pH < 2 by adding 1 mL of concentrated HCl per 100 gram of sample and cooled to 4 ± 2 °C. If acidification is not practical (as with a dry soil), the addition of the HCl is not required and the sample should be cooled to 4 ± 2 °C. The laboratory must be notified so that the sample can be acidified prior to analysis.

8.3 A holding time has not been established for HEM in solids, but it is recommended that the sample be analyzed as soon as possible.

9.0 QUALITY CONTROL

9.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated.

9.2 Employ a minimum of one method blank per analytical batch or twenty samples, whichever is more frequent, to verify that all reagents, solvents, and equipment are contamination free. Prepare the method blank from 5 g of inert matrix such as pre-cleaned sand or similar material, and carry it through the analytical process.

9.3 Run one matrix duplicate and matrix spike sample every twenty samples or analytical batch, whichever is more frequent. Matrix duplicates and spikes are brought through the whole sample preparation and analytical process.

9.4 The performance of the method should be evaluated by the use of a Laboratory Control Sample (LCS). The LCS is prepared by spiking an inert matrix (as pre-cleaned sand or similar material) with an appropriate volume of spiking solution (Sec. 7.7) and carrying it through the analytical process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibrate the analytical balance at 2 mg and 1000 mg using class "S" weights.

10.2 Calibration shall be within $\pm 10\%$ (i.e., ± 0.2 mg) at 2 mg and $\pm 0.5\%$ (i.e., ± 5 mg) at 1000 mg. If values are not within these limits, recalibrate the balance.

11.0 PROCEDURE

11.1 Determination of Sample Dry Weight Fraction

11.1.1 When it is necessary to report the HEM on a dry weight basis, determine the dry weight fraction using a separate aliquot of sample, as discussed below. The aliquot used for this determination cannot be used to evaluate HEM.

11.1.2 Weigh 5-10 gram (\pm 0.01 gram) of the sample into pre-weighed crucible. Determine the weight of the wet sample by subtracting the weight of the crucible.

11.1.3 Place the crucible with the wet sample in an oven overnight at 105°C. Remove crucible from oven and place in a desiccator to cool. Weigh. Determine dry weight of sample by subtracting the weight of the crucible. Determine the dry weight fraction of the sample as follows:

NOTE: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

$$\text{dry weight fraction} = \frac{\text{g of dry sample}}{\text{g of sample}}$$

11.2 Sample Preparation

11.2.1 Sludge/Waste Samples

11.2.1.1 Weigh out 20 ± 0.5 grams of wet sample into a 150-mL beaker.

11.2.1.2 If the sample has not been acidified, acidify to a pH \leq 2 with approximately 0.3 mL concentrated HCl.

11.2.1.3 Add 25 grams $\text{Mg}_2\text{SO}_4 \cdot \text{H}_2\text{O}$ (Sec. 7.4) and stir to a smooth paste.

11.2.1.4 Spread paste on sides of beaker to facilitate evaporation. Let stand about 15-30 min or until material is solidified.

11.2.1.5 Remove solids and grind to fine powder in a mortar.

11.2.1.6 Add the powder to the paper extraction thimble.

11.2.1.7 Wipe beaker and mortar with pieces of filter paper moistened with n-hexane and add to thimble.

11.2.1.8 Fill thimble with glass wool (or glass beads).

11.2.2 Sediment/Soil Samples

11.2.2.1 Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

11.2.2.2 Blend 10 grams of the sample with 10 grams of anhydrous sodium sulfate (Sec. 7.5) as described in Section 11.2.1. Transfer homogenized paste to an extraction thimble and cover with glass wool or glass beads. The extraction thimble must drain freely for the duration of the extraction period.

11.3 Extraction

11.3.1 Set-up the Soxhlet apparatus containing the extraction thimble and sample and attach a 125-mL boiling flask containing 90 mL of n-hexane. Add boiling chips. Adjust the heating control on the heating mantle so that a cycling rate of 20 cycles/h is obtained. Extract for a period of 4 hrs.

11.3.2 Tare a clean 250-mL or appropriate sized boiling flask as follows:

11.3.2.1 Dry the flask in an oven at 105-115°C for a minimum of 2 h.

11.3.2.2 Remove from the oven and immediately transfer to a desiccator to cool at room temperature.

11.3.2.3 When cool, remove from the desiccator with tongs and weigh immediately on a calibrated balance.

11.3.3 At the end of the 4 h extraction period, filter the extract through grease-free cotton, into the pre-weighed boiling flask (Sec. 11.3.2). Use gloves to avoid adding fingerprints to the flask.

11.3.4 Rinse flask and cotton with n-hexane and add to the 250-mL boiling flask.

NOTE: If the extract is clear and no suspended particles are present, the filtration step may be omitted.

11.3.5 Connect the boiling flask to the distilling head apparatus and distill the solvent by immersing the lower half of the flask in a water bath or a steam bath. A heating mantle may also be used. Adjust the temperature of the heating device to complete the distillation in less than 30 minutes. Collect the solvent for reuse or appropriate disposal.

11.3.6 When the distillation is complete, remove the distilling head. Immediately remove the flask from the heat source and wipe the outside to remove excess moisture and fingerprints. To remove solvent vapor, sweep out the flask for 15 sec with air by inserting a glass tube that is connected to a vacuum source.

11.3.7 Cool the boiling flask in a desiccator for 30 min and weigh. Determine the gain in weight of the boiling flask by subtracting the weight of the boiling flask (Sec. 11.3.2) from the final boiling flask weight.

12.0 DATA ANALYSIS AND CALCULATIONS

Calculate the concentration of HEM in the sample as follows:

$$\text{HEM (mg/kg wet weight)} = \frac{\text{gain in weight of flask(mg)} \times 1000}{\text{weight of wet solid(g)}}$$

NOTE:

If it is necessary to report the results on a dry weight basis, divide the result obtained above by the dry weight fraction calculated in Sec. 11.1.3. Report the results as mg/kg HEM dry weight. If it is necessary to report the results as a percentage of the wet or dry weight, divide the wet-weight concentration or dry weight concentration by 10,000 and report the result as % HEM wet or dry weight.

13.0 METHOD PERFORMANCE

In a preliminary study designed to find a suitable replacement for Freon-113, three EPA contract laboratories evaluated a total of 28 solid samples derived from various industrial and commercial processes for oil and grease. This study evaluated a total of six solvents, including n-hexane, to determine which of the alternative solvents produced results most closely with that of Freon-113. In this study, each waste was Soxhlet-extracted in triplicate using Freon-113 and each of the alternative solvents. Based on the overall results, n-hexane was judged to be the best alternative solvent. The data provided in Table 1 compare the results for Freon-113 and n-hexane for each waste. For a complete discussion of this study, refer to reference 1 in Section 16.0.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable Federal, state and local rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer

discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Preliminary Report of EPA Efforts to Replace Freon for the Determination of Oil and Grease, United States Environmental Protection Agency, Office of Water, EPA-821-93-009. June 1993.
2. Method 1664, Revision A: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The pages to follow contain Table 1, and a flow diagram of the method procedure.

TABLE 1

SOXHLET EXTRACTION OF SOLIDS USING FREON-113 AND N-HEXANE
All concentrations in mg/kg

Facility/ Process	Waste Stream	Solvent: Freon Hexane	Rep No. 1	Rep No. 2	Rep No. 3	Mean Concen- tration	Standard Deviation
Paper Mill	Dewatered Sludge	Freon Hexane	11000	5300	7900	8000	2762
			6600	2400	11000	6600	4203
POTW	Sewage Sludge	Freon Hexane	98000	81000	81000	87000	9940
			110000	86000	80000	91000	13281
Leather Tannery	Dewatered Sludge	Freon Hexane	11000	12000	12000	12000	732
			21000	15000	19000	18000	3201
POTW	Digested Sludge	Freon Hexane	130000	97000	66000	98000	33028
			54000	76000	48000	59000	14516
Petroleum Refinery	API Separator Sludge	Freon Hexane	320000	350000	250000	310000	53257
			240000	320000	240000	270000	43822
Industrial Laundry	DAF Sludge	Freon Hexane	310000	310000	240000	290000	41717
			290000	360000	180000	280000	90819
Fish Oil Plant	Oily Sludge	Freon Hexane	890000	1000000	770000	890000	131249
			440000	530000	460000	480000	46318
Coke Plant	Waste Activated Sludge	Freon Hexane	8300	8000	18000	11000	5505
			14000	19000	15000	16000	2732
Wood Preserving Plant	Solid Waste	Freon Hexane	150000	140000	140000	140000	3512
			140000	130000	130000	130000	6557
Drilling Fluid Supplier	Used drilling mud	Freon Hexane	1300	1600	1300	1400	157
			1300	1200	1600	1400	201
Contam. Soils	Kerosene Contaminated Soil	Freon Hexane	2000	1400	1900	1700	352
			2500	3200	2600	2800	410
Poultry Plant	Waste Activated Sludge	Freon Hexane	38000	11000	40000	30000	16263
			5900	11000	46000	21000	21795
Rolling Mill	Dewatered Scale	Freon Hexane	11000	14000	17000	14000	2884
			14000	14000	16000	15000	983
Mayonnaise Plant	Oily Sludge	Freon Hexane	880000	850000	780000	840000	50521
			590000	780000	520000	630000	132020
Seafood Plant	Waste Sludge	Freon Hexane	64000		53000	58000	7526
			34000	31000	27000	31000	3867

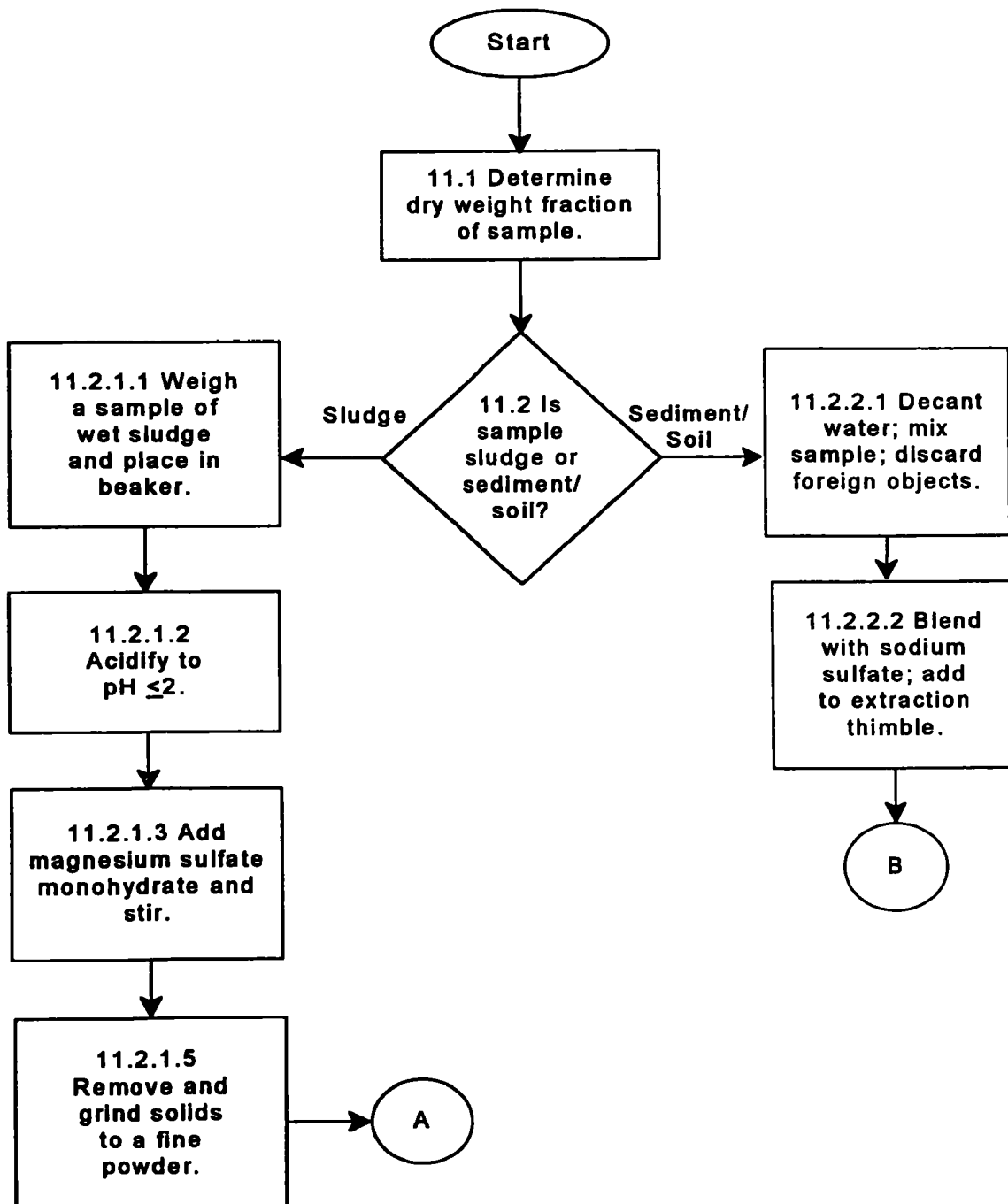
TABLE 1
(CONTINUED)

Facility/ Process	Waste Stream	Solvent: Freon Hexane	Rep No. 1	Rep No. 2	Rep No. 3	Mean Concen- tration	Standard Deviation
Seafood Plant	Oily Sludge	Freon Hexane	400000	410000	430000	410000	16371
			400000	390000	390000	400000	7095
Poultry Plant	DAF Sludge	Freon Hexane	670000	600000	570000	610000	49549
			530000	530000	530000	530000	2449
Railroad Yard	Oily Sludge	Freon Hexane	870000	920000	870000	890000	27906
			850000	840000	830000	840000	6884
Can Manufact Plant	Filter Cake	Freon Hexane	62000	62000	60000	61000	976
			69000	64000	66000	66000	2615
Soup Plant	DAF Sludge	Freon Hexane	600000	590000	610000	600000	10066
			580000	520000	600000	570000	40361
Oily Water Treatment Plant	Oily Sludge	Freon Hexane	76000	75000	70000	74000	3215
			77000	60000	79000	72000	10713
Can Manufact Plant	Oily Sludge	Freon Hexane	94000	88000	94000	92000	3291
			80000	90000	83000	85000	4992
Can Manufact Plant	Filter Cake	Freon Hexane	290000	290000	300000	290000	6217
			290000	290000	290000	290000	2029
Drum Handling Facility	Oily Sludge	Freon Hexane	1200000	1100000	1200000	1200000	57735
			990000	1000000	980000	1000000	27319
Polymer Plant	Dewatered Sludge	Freon Hexane	13000	12000	8200	11000	2524
			8400	6900	9100	8100	1122
Restaurant	Vegetable Oil Waste	Freon Hexane	760000	610000	780000	720000	92060
			1100000	980000	980000	1000000	80064
Leather Tannery	Waste Sludge	Freon Hexane	180000	220000	190000	190000	22140
			240000	270000	210000	240000	31177

Source: Reference 1

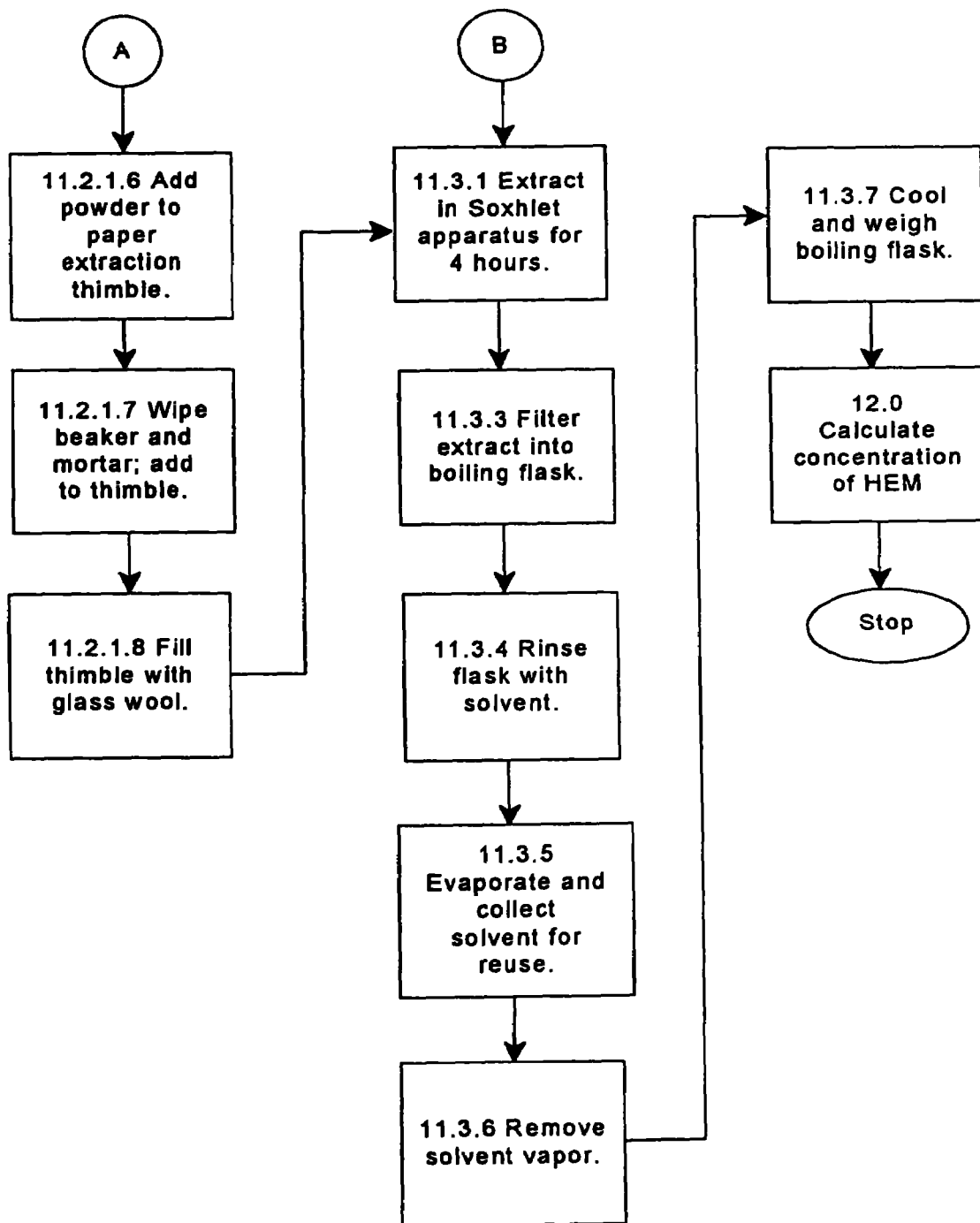
METHOD 9071B

n-HEXANE EXTRACTABLE MATERIAL (HEM) FOR SLUDGE, SEDIMENT, AND SOLID SAMPLES



METHOD 9071B

n-HEXANE EXTRACTABLE MATERIAL (HEM) FOR SLUDGE, SEDIMENT, AND SOLID SAMPLES
(Continued)



CHAPTER SIX

PROPERTIES

Prior to employing the methods in this chapter, analysts are advised to consult the disclaimer statement at the front of this manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. Procedures for required method-defined parameters are not subject to the same flexibility afforded in other SW-846 methods. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

The following methods are found in Chapter Six:

Method 1030:	Ignitability of Solids
Method 1120:	Dermal Corrosion
Method 1312:	Synthetic Precipitation Leaching Procedure
Method 1320:	Multiple Extraction Procedure
Method 1330A:	Extraction Procedure for Oily Wastes
Method 9041A:	pH Paper Method
Method 9045D:	Soil and Waste pH
Method 9050A:	Specific Conductance
Method 9080:	Cation-Exchange Capacity of Soils (Ammonium Acetate)
Method 9081:	Cation-Exchange Capacity of Soils (Sodium Acetate)
Method 9090A:	Compatibility Test for Wastes and Membrane Liners
Method 9095B:	Paint Filter Liquids Test
Method 9096:	Liquid Release Test (LRT) Procedure
Appendix A:	Liquid Release Test Pre-Test
Method 9100:	Saturated Hydraulic Conductivity, Saturated Leachate Conductivity, and Intrinsic Permeability
Method 9310:	Gross Alpha and Gross Beta
Method 9315:	Alpha-Emitting Radium Isotopes

METHOD 9045D

SOIL AND WASTE pH

1.0 SCOPE AND APPLICATION

1.1 This method is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

2.0 SUMMARY OF METHOD

2.1 The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors

3.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.0 APPARATUS AND MATERIALS

4.1 pH meter with means for temperature compensation.

4.2 Glass electrode.

4.3 Reference electrode -- A silver-silver chloride or other reference electrode of constant potential may be used.

NOTE: Combination electrodes incorporating both measuring and referenced functions are convenient to use and are available with solid, gel-type filling materials that require minimal maintenance.

4.4 Beaker -- 50-mL.

4.5 Thermometer and/or temperature sensor for automatic compensation.

4.6 Analytical balance -- capable of weighing 0.1 g

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

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

5.4 Secondary standard buffers may be prepared from NIST salts or purchased as solutions from commercial vendors. These commercially available solutions, which have been validated by comparison with NIST standards, are recommended for routine use.

6.0 SAMPLE PRESERVATION AND HANDLING

Samples should be analyzed as soon as possible.

7.0 PROCEDURE

7.1 Calibration

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

7.1.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value. If an accurate pH reading based on the conventional pH scale [0 to 14 at 25 °C] is required, the analyst should control sample temperature at 25 ± 1 °C when sample pH approaches the alkaline end of the scale (e.g., a pH of 11 or above).

7.2 Sample preparation and pH measurement of soils:

7.2.1 To 20 g of soil in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 min. Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices.

7.2.2 Let the soil suspension stand for about 1 hr to allow most of the suspended clay to settle out from the suspension or filter or centrifuge off the aqueous phase for pH measurement.

7.2.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrodes into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

7.2.4 If the sample temperature differs by more than 2 °C from the buffer solution, the measured pH values must be corrected.

7.2.5 Report the results as "soil pH measured in water at __ °C" where "__ °C" is the temperature at which the test was conducted.

7.3 Sample preparation and pH measurement of waste materials

7.3.1 To 20 g of waste sample in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 min. Additional dilutions are allowed if working with hygroscopic wastes and salts or other problematic matrices.

7.3.2 Let the waste suspension stand for about 15 min to allow most of the suspended waste to settle out from the suspension or filter or centrifuge off aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 mL of reagent water

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Step 3.3) if it becomes coated with an oily material.

7.3.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant to establish good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrode into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

7.3.4 If the sample temperature differs by more than 2 °C from the buffer solution, the measured pH values must be corrected.

7.3.5 Report the results as "waste pH measured in water at __ °C" where "__ °C" is the temperature at which the test was conducted.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for the appropriate QC protocols.

8.2 Electrodes must be thoroughly rinsed between samples.

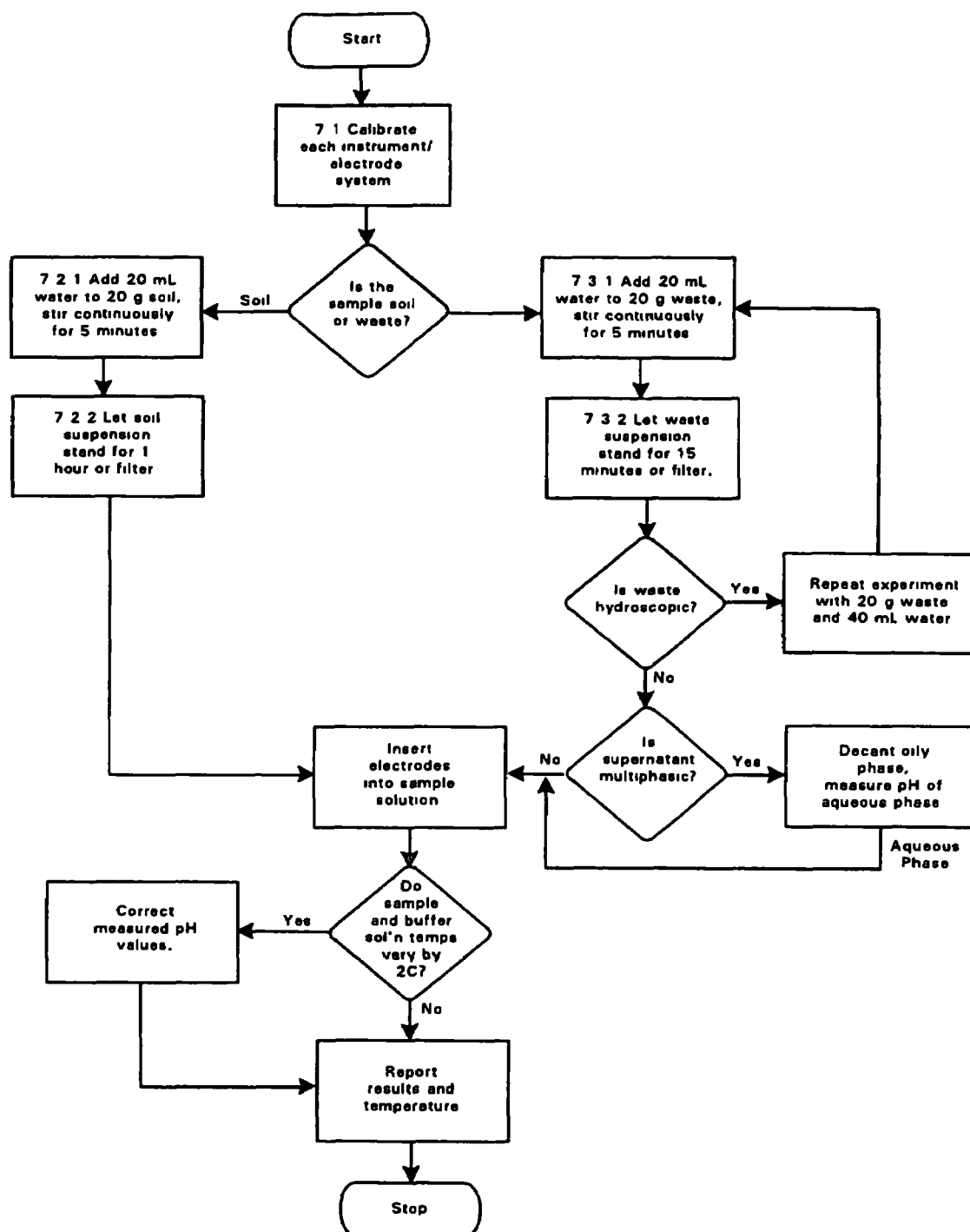
9.0 METHOD PERFORMANCE

9.1 No data provided.

10.0 REFERENCES

1. Black, Charles Allen; Methods of Soil Analysis; American Society of Agronomy: Madison, WI, 1973.
2. National Bureau of Standards, Standard Reference Material Catalog, 1986-87, Special Publication 260.

METHOD 9045D
SOIL AND WASTE pH



METHOD 9095B

PAINT FILTER LIQUIDS TEST

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the presence of free liquids in a representative sample of waste.

1.2 The method is used to determine compliance with 40 CFR 264.314 and 265.314.

2.0 SUMMARY OF METHOD

2.1 A predetermined amount of material is placed in a paint filter. If any portion of the material passes through and drops from the filter within the 5-min test period, the material is deemed to contain free liquids.

3.0 INTERFERENCES

3.1 Filter media were observed to separate from the filter cone on exposure to alkaline materials. This development causes no problem if the sample is not disturbed.

3.2 Temperature can affect the test results if the test is performed below the freezing point of any liquid in the sample. Tests must be performed above the freezing point and can, but are not required to, exceed room temperature of 25 °C.

4.0 APPARATUS AND MATERIALS

4.1 Conical paint filter -- Mesh number 60 +/- 5% (fine meshed size) Available at local paint stores such as Sherwin-Williams and Glidden

4.2 Glass funnel -- If the paint filter, with the waste, cannot sustain its weight on the ring stand, then a fluted glass funnel or glass funnel with a mouth large enough to allow at least 1 in. of the filter mesh to protrude should be used to support the filter. The funnel should be fluted or have a large open mouth in order to support the paint filter yet not interfere with the movement, to the graduated cylinder, of the liquid that passes through the filter mesh.

4.3 Ring stand and ring, or tripod.

4.4 Graduated cylinder or beaker -- 100-mL

5.0 REAGENTS

5.1 None

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

A 100-mL or 100-g representative sample is required for the test. If it is not possible to obtain a sample of 100 mL or 100 g that is sufficiently representative of the waste, the analyst may use larger size samples in multiples of 100 mL or 100 g, i.e., 200, 300, 400 mL or g. However, when larger samples are used, analysts shall divide the sample into 100-mL or 100-g portions and test each portion separately. If any portion contains free liquids, the entire sample is considered to have free liquids. If the sample is measured volumetrically, then it should lack major air spaces or voids.

7.0 PROCEDURE

7.1 Assemble test apparatus as shown in Figure 1.

7.2 Place sample in the filter. A funnel may be used to provide support for the paint filter. If the sample is of such light bulk density that it overflows the filter, then the sides of the filter can be extended upward by taping filter paper to the inside of the filter and above the mesh. Settling the sample into the paint filter may be facilitated by lightly tapping the side of the filter as it is being filled.

7.3 In order to assure uniformity and standardization of the test, material such as sorbent pads or pillows which do not conform to the shape of the paint filter should be cut into small pieces and poured into the filter. Sample size reduction may be accomplished by cutting the sorbent material with scissors, shears, a knife, or other such device so as to preserve as much of the original integrity of the sorbent fabric as possible. Sorbents enclosed in a fabric should be mixed with the resultant fabric pieces. The particles to be tested should be reduced smaller than 1 cm (i.e., should be capable of passing through a 9.5 mm (0.375 inch) standard sieve). Grinding sorbent materials should be avoided as this may destroy the integrity of the sorbent and produce many "fine particles" which would normally not be present.

7.4 For brittle materials larger than 1 cm that do not conform to the filter, light crushing to reduce oversize particles is acceptable if it is not practical to cut the material. Materials such as clay, silica gel, and some polymers may fall into this category.

7.5 Allow sample to drain for 5 min into the graduated cylinder.

7.6 If any portion of the test material collects in the graduated cylinder in the 5-min period, then the material is deemed to contain free liquids for purposes of 40 CFR 264.314 and 265.314.

8.0 QUALITY CONTROL

8.1 Duplicate samples should be analyzed on a routine basis.

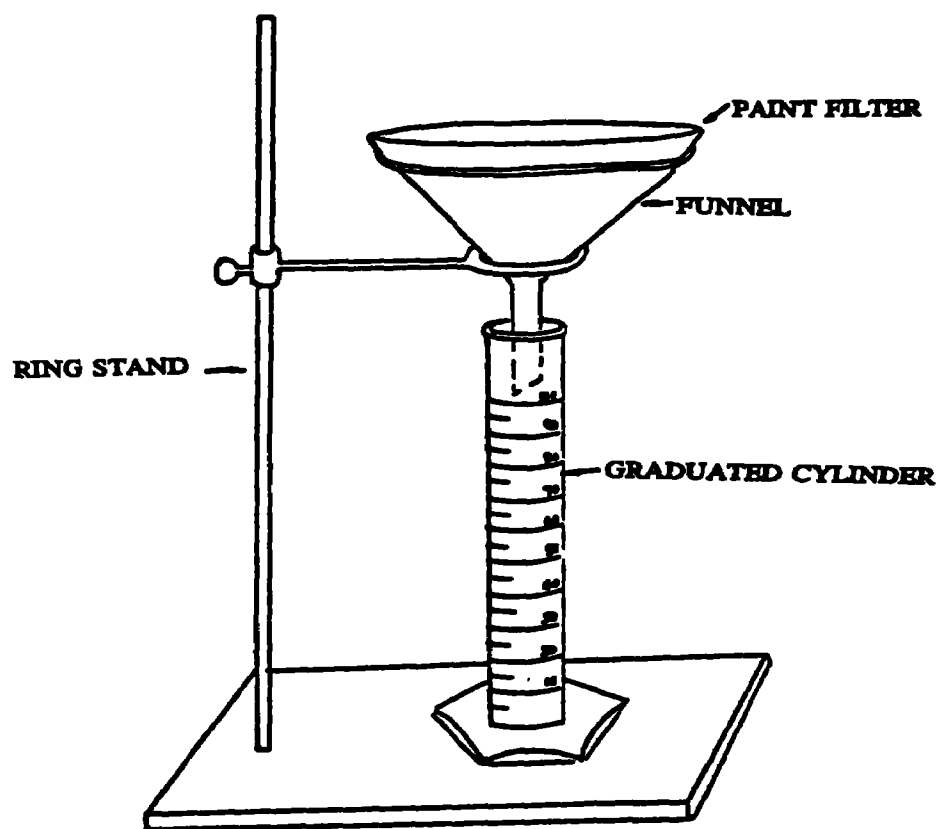
9.0 METHOD PERFORMANCE

9.1 No data provided.

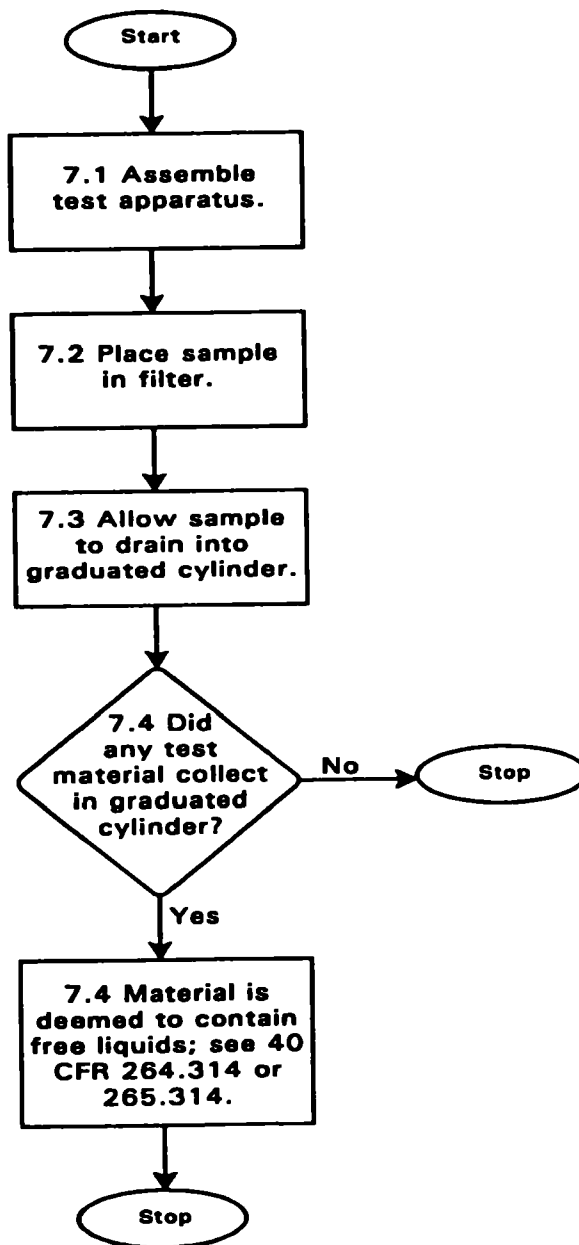
10.0 REFERENCES

10.1 None provided.

FIGURE 1
PAINT FILTER TEST APPARATUS



METHOD 9095B
PAINT FILTER LIQUIDS TEST



CHAPTER SEVEN

CHARACTERISTICS INTRODUCTION AND REGULATORY DEFINITIONS

This chapter addresses procedures for required "method-defined parameters," where the analytical result is wholly dependant on the process used to make the measurement. Examples include the use of the toxicity characteristic leaching procedure (TCLP) to prepare a leachate, and the flash point, pH, paint filter liquids, and corrosivity tests. In these instances, changes to the specific methods may change the end result and incorrectly identify a waste as nonhazardous. Therefore, when the measurement of such method-defined parameters is required by regulation, those methods are not subject to the flexibility afforded in other SW-846 methods (such as described in the Disclaimer and Chapter Two of this manual).

7.1 IGNITABILITY

7.1.1 Introduction

The objective of the ignitability characteristic is to identify wastes that either present fire hazards under routine storage, disposal, and transportation or are capable of severely exacerbating a fire once started.

7.1.2 Regulatory Definition

See 40 CFR 261.21 for the regulatory definition of the hazardous waste characteristic of ignitability. Methods 1010 and 1020 of Chapter Eight refer the reader to the ASTM standards required by the RCRA regulations for the flash point of liquids at 40 CFR 261.21(1)

7.2 CORROSIVITY

7.2.1 Introduction

The corrosivity characteristic, as defined in 40 CFR 261.22, is designed to identify wastes that might pose a hazard to human health or the environment due to their ability to:

1. Mobilize toxic metals if discharged into a landfill environment;
2. Corrode handling, storage, transportation, and management equipment, or
3. Destroy human or animal tissue in the event of inadvertent contact.

In order to identify such potentially hazardous materials, EPA has selected two properties upon which to base the definition of a corrosive waste. These properties are pH and corrosivity toward Type SAE 1020 steel.

The procedures for measuring pH of aqueous wastes are detailed in Method 9040, Chapter Six. Method 1110, Chapter Eight, describes how to determine whether a waste is corrosive to steel. Use Method 9095, Paint Filter Liquids Test, Chapter Six, to determine free liquid.

7.2.2 Regulatory Definition

See 40 CFR 261.22 for the regulatory definition of the hazardous waste characteristic of corrosivity

7.3 REACTIVITY

7.3.1 Introduction

The regulation in 40 CFR 261.23 defines reactive wastes to include wastes that have any of the following properties. (1) readily undergo violent chemical change; (2) react violently or form potentially explosive mixtures with water; (3) generate toxic fumes when mixed with water or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic or basic conditions; (4) explode when subjected to a strong initiating force; (5) explode at normal temperatures and pressures, or (6) fit within the Department of Transportation's forbidden explosives, Class A explosives, or Class B explosives classifications.

This definition is intended to identify wastes that, because of their extreme instability and tendency to react violently or explode, pose a problem at all stages of the waste management process. The Agency relies entirely on a descriptive, prose definition of reactivity because available tests for measuring the variegated class of effects embraced by the reactivity definition suffer from a number of deficiencies.

7.3.2 Regulatory Definition

See 40 CFR 261.24 for the regulatory definition of the hazardous waste characteristic of reactivity.

7.4 TOXICITY CHARACTERISTIC LEACHING PROCEDURE

7.4.1 Introduction

The Toxicity Characteristic Leaching Procedure (TCLP) is designed to simulate the leaching a waste will undergo if disposed of in a sanitary landfill. This test is designed to simulate leaching that takes place in a sanitary landfill only. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A subsample of a waste is extracted with the appropriate buffered acetic acid solution for 18 ± 2 hours. The extract obtained from the TCLP (the "TCLP extract") is then analyzed to determine if any of the thresholds established for the 40 Toxicity Characteristic (TC) constituents (listed in Table 7-1) have been exceeded or if the treatment standards established for the constituents listed in 40 CFR 268.40 have been met under the Land Disposal Restrictions (LDR) regulations. If the TCLP extract contains any one of the TC constituents in an amount equal to or exceeding the concentrations specified in 40 CFR 261.24, the waste possesses the characteristic of toxicity and is a hazardous waste. If the TCLP extract contains constituents in an amount exceeding the concentrations specified in 40 CFR 268.40, the treatment standard for that waste has not been met, and further treatment is necessary prior to land disposal.

7.4.2 Summary of Procedure

Figure 3 summarizes the procedures in the TCLP. The five basic steps of the TCLP are summarized below

1. Separation Procedure

For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis.

2. Particle Size Reduction

Prior to extraction, the solid material must pass through a 9.5-mm (0.375-in.) standard sieve, have a surface area per gram of material equal to or greater than 3.1 cm^2 , or, be smaller than 1 cm in its narrowest dimension. If the surface area is smaller or the particle size larger than described above, the solid portion of the waste is prepared for extraction by crushing, cutting, or grinding the waste to the surface area or particle size described above. (Special precautions must be taken if the solids are prepared for organic volatiles extraction.)

3. Extraction of Solid Material

The solid material from Step 2 is extracted for 18 ± 2 hours with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes.

4. Final Separation of the Extraction from the Remaining Solid

Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter. If compatible, the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

5. Testing (Analysis) of TCLP Extract

Inorganic and organic species are identified and quantified using appropriate methods in the 6000, 7000, and 8000 series of methods in this manual or by other appropriate methods.

7.4.3 Regulatory Definition

Under the Toxicity Characteristic, a solid waste exhibits the characteristic of toxicity if the TCLP extract from a subsample of the waste contains any of the contaminants listed in Table 7-1 at a concentration greater than or equal to the respective value given in that table. If a waste contains <0.5% filterable solids, the waste itself, after filtering, is considered to be the extract for the purposes of analysis.

Under the Land Disposal Restrictions regulations (40 CFR, Part 268), a restricted waste identified in 40 CFR 268.40 cannot be land disposed if a TCLP extract of the waste or a TCLP extract of the treatment residue of the waste exceeds the values shown in the table of 40 CFR 268.40 for any hazardous constituent listed in the table for that waste. If a waste contains <0.5% filterable solids, the waste itself, after filtering, is considered to be the extract for the purposes of analysis.

TABLE 7-1.
MAXIMUM CONCENTRATION OF CONTAMINANTS FOR TOXICITY CHARACTERISTIC

Contaminant	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresol	200.0 ¹
m-Cresol	200.0 ¹
p-Cresol	200.0 ¹
Cresol	200.0 ¹
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
1,1-Dichloroethylene	0.7
2,4-Dinitrotoluene	0.13 ²
Endrin	0.02
Heptachlor (and its hydroxide)	0.008
Hexachlorobenzene	0.13 ²
Hexachloro-1,3-butadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0 ²
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

¹If o-, m-, and p-cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level of total cresol is 200 mg/L.

²Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

FIGURE 3.

TOXICITY CHARACTERISTIC LEACHING PROCEDURE FLOWCHART

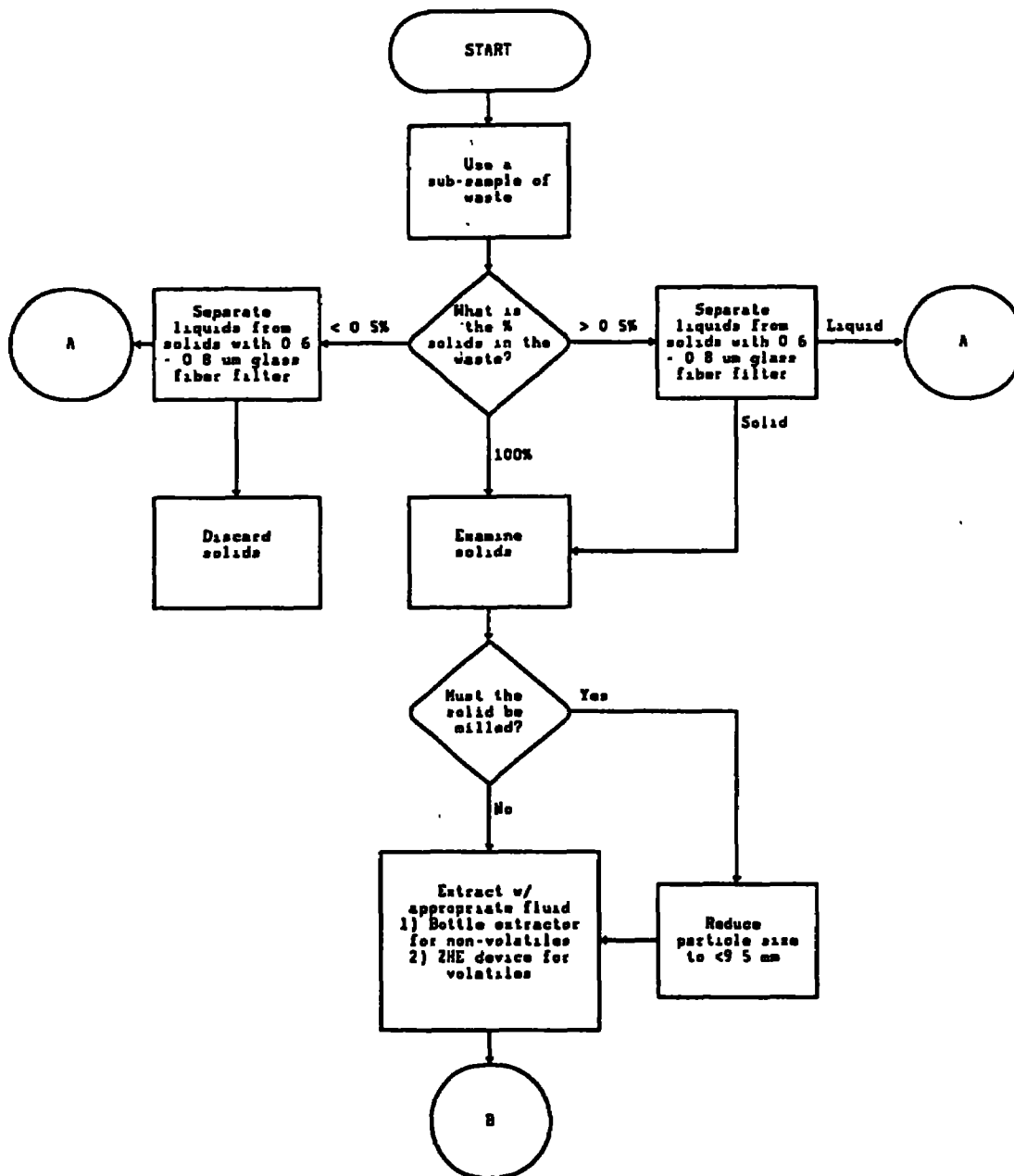
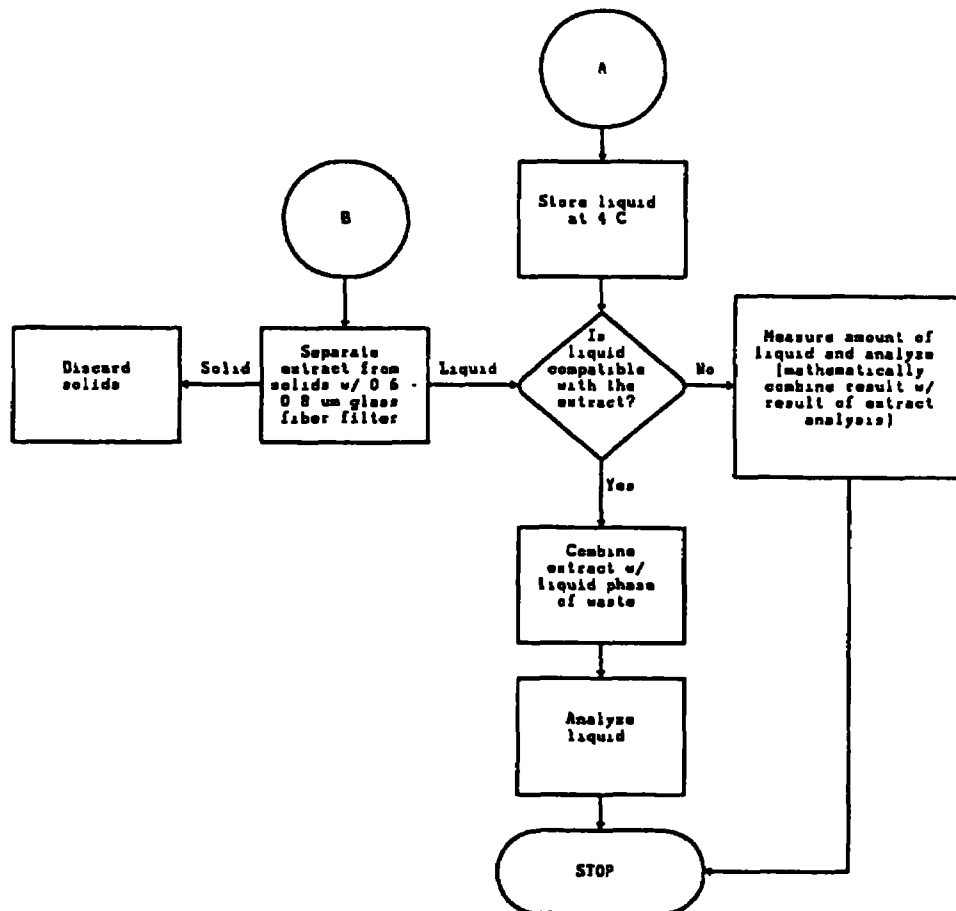


FIGURE 3 (continued)



CHAPTER EIGHT

METHODS FOR DETERMINING CHARACTERISTICS

This chapter addresses procedures for required method-defined parameters, where the analytical result is wholly dependant on the process used to make the measurement. Examples include the use of the toxicity characteristic leaching procedure (TCLP) to prepare a leachate, and the flash point, pH, paint filter liquids, and corrosivity tests. In these instances, changes to the specific methods may change the end result and incorrectly identify a waste as nonhazardous. Therefore, when the measurement of such method-defined parameters is required by regulation, those methods are not subject to the flexibility afforded in other SW-846 methods (such as described in the Disclaimer and Chapter Two of this manual).

Methods for determining the characteristics of ignitability for liquids, corrosivity for liquids, and toxicity are included. The text of the methods identified for the characteristic of ignitability refer the reader to the appropriate required ASTM methods. There are no required SW-846 methods for the analysis of the characteristic of reactivity.

8.1 Ignitability

This chapter addresses procedures for required method-defined parameters, where the analytical result is wholly dependant on the process used to make the measurement. Examples include the use of the toxicity characteristic leaching procedure (TCLP) to prepare a leachate, and the flash point, pH, paint filter liquids, and corrosivity tests. In these instances, changes to the specific methods may change the end result and incorrectly identify a waste as nonhazardous. Therefore, when the measurement of such method-defined parameters is required by regulation, those methods are not subject to the flexibility afforded in other SW-846 methods (such as described in the Disclaimer and Chapter Two of this manual).

The text of the methods identified for the characteristic of ignitability refer the reader to the appropriate required ASTM methods. The following methods are found in Sec. 8.1 of this chapter:

Method 1010A:	Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
Method 1020B :	Standard Test Methods for Flash Point by Setaflash (Small Scale) Closed-cup Apparatus

METHOD 1010A

TEST METHODS FOR FLASH POINT BY PENSKEY-MARTENS CLOSED CUP TESTER

See American Society for Testing Materials (ASTM) Standard D 93-79 or Standard D 93-80 for these method procedures used in the characterization of flash point of liquids. These standards are two of three required method options for determination of the ignitability characteristic in liquid wastes, see 40 CFR 261.21(a)(1). Currently, these ASTM standards can be obtained from Global Engineering Documents, 15 Iverness Way East, Englewood, CO 80112, 1-800-854-7179, <http://global.ihs.com>. At the web site, do a search for "ASTM D 93" (document number) and at the document search results select "view historical revisions." Select the Revision 79 or 80 version.

METHOD 1020B

STANDARD TEST METHODS FOR FLASH POINT BY SETAFLASH (SMALL SCALE) CLOSED-CUP APPARATUS

See American Society for Testing Materials (ASTM) Standard D 3278-78 for these method procedures used in the characterization of the flash point of liquids. This standard is one of three required method options for determination of the ignitability characteristic in liquid wastes, see 40 CFR 261.21(a)(1). Currently, this ASTM standard can be obtained from Global Engineering Documents, 15 Iverness Way East, Englewood, CO 80112, 1-800-854-7179, <http://global.ihs.com>. At the web site, do a search for "ASTM D 3278" (document number) and at the document search results select "view historical revisions." Select the Revision 78 version.

8.2 Corrosivity

This chapter addresses procedures for required method-defined parameters, where the analytical result is wholly dependant on the process used to make the measurement. Examples include the use of the toxicity characteristic leaching procedure (TCLP) to prepare a leachate, and the flash point, pH, paint filter liquids, and corrosivity tests. In these instances, changes to the specific methods may change the end result and incorrectly identify a waste as nonhazardous. Therefore, when the measurement of such method-defined parameters is required by regulation, those methods are not subject to the flexibility afforded in other SW-846 methods (such as described in the Disclaimer and Chapter Two of this manual).

The following methods are found in Sec. 8.2 of this chapter:

Method 9040C:	pH Electrometric Measurement
Method 1110A:	Corrosivity Toward Steel

METHOD 9040C

pH ELECTROMETRIC MEASUREMENT

1.0 SCOPE AND APPLICATION

1.1 This method is used to measure the pH of aqueous wastes and those multiphase wastes where the aqueous phase constitutes at least 20% of the total volume of the waste

1.2 The corrosivity of concentrated acids and bases, or of concentrated acids and bases mixed with inert substances, cannot be measured. The pH measurement requires some water content.

2.0 SUMMARY

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

3.0 INTERFERENCES

3.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or moderate (<0.1 molar solution) salinity.

3.2 Sodium error at pH levels >10 can be reduced or eliminated by using a low-sodium-error electrode.

3.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1.10) may be necessary to remove any remaining film.

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

4.0 APPARATUS AND MATERIALS

4.1 pH meter -- Laboratory or field model. Many instruments are commercially available with various specifications and optional equipment.

4.2 Glass electrode.

4.3 Reference electrode -- A silver-silver chloride or other reference electrode of constant potential may be used.

NOTE Combination electrodes incorporating both measuring and referenced functions are convenient to use and are available with solid, gel-type filling materials that require minimal maintenance.

4.4 Magnetic stirrer and Teflon-coated stirring bar

4.5 Thermometer and/or temperature sensor for automatic compensation

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

5.3 Secondary standard buffers may be prepared from NIST salts or purchased as solutions from commercial vendors. These commercially available solutions have been validated by comparison with NIST standards and are recommended for routine use.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Samples should be analyzed as soon as possible.

7.0 PROCEDURE

7.1 Calibration

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

7.1.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. (For corrosivity characterization, the calibration of the pH meter should include a buffer of pH 2 for acidic wastes and a pH 12 buffer for caustic wastes; also, for corrosivity characterization, the sample must be measured at 25 ± 1 °C if the pH of the waste is above 12.0.) Various instrument designs may involve use of a dial (to "balance" or "standardize") or a slope adjustment, as outlined in the manufacturer's instructions. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value.

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

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

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

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for the appropriate QC protocols.

8.2 Electrodes must be thoroughly rinsed between samples.

9.0 METHOD PERFORMANCE

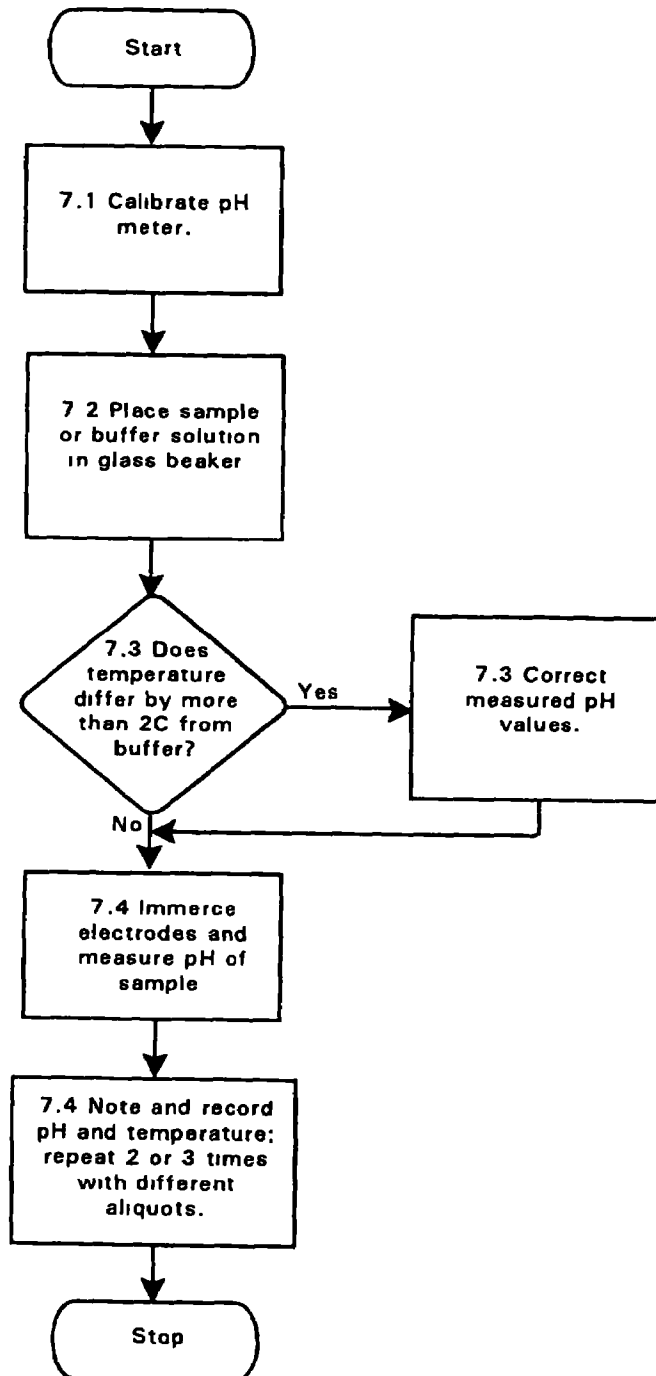
9.1 Forty-four analysts in twenty laboratories analyzed six synthetic water samples containing exact increments of hydrogen-hydroxyl ions, with the following results:

pH Units	Standard Deviation pH Units	Accuracy as	
		Bias %	Bias pH Units
3.5	0.10	-0.29	-0.01
3.5	0.11	-0.00	
7.1	0.20	+1.01	+0.07
7.2	0.18	-0.03	-0.002
8.0	0.13	-0.12	-0.01
8.0	0.12	+0.16	+0.01

10.0 REFERENCES

- 1. National Bureau of Standards, Standard Reference Material Catalog 1986-87, Special Publication 260.**

METHOD 9040C
pH ELECTROMETRIC MEASUREMENT



METHOD 1110A

CORROSIVITY TOWARD STEEL

1.0 SCOPE AND APPLICATION

1.1 This method measures the corrosivity toward steel of both aqueous and nonaqueous liquid wastes.

2.0 SUMMARY OF METHOD

2.1 This test exposes coupons of SAE Type 1020 steel to the liquid waste to be evaluated and, by measuring the degree to which the coupon has been dissolved, determines the corrosivity of the waste.

3.0 INTERFERENCES

3.1 In laboratory tests, such as this one, corrosion of duplicate coupons is usually reproducible to within 10%. However, large differences in corrosion rates may occasionally occur under conditions where the metal surfaces become passivated. Therefore, at least duplicate determinations of corrosion rate should be made.

4.0 APPARATUS AND MATERIALS

4.1 An apparatus should be used, consisting of a kettle or flask of suitable size (usually 500 to 5,000 mL), a reflux condenser, a thermowell and temperature regulating device, a heating device (mantle, hot plate, or bath), and a specimen support system. A typical resin flask set up for this type of test is shown in Figure 1.

4.2 The supporting device and container shall be constructed of materials that are not affected by, or cause contamination of, the waste under test.

4.3 The method of supporting the coupons will vary with the apparatus used for conducting the test, but it should be designed to insulate the coupons from each other physically and electrically and to insulate the coupons from any metallic container or other device used in the test. Some common support materials include glass, fluorocarbon, or coated metal.

4.4 The shape and form of the coupon support should ensure free contact with the waste.

4.5 A circular specimen of SAE 1020 steel of about 3.75 cm (1.5 in.) diameter is a convenient shape for a coupon. With a thickness of approximately 0.32 cm (0.125 in.) and a 0.80-cm (0.4-in.)-diameter hole for mounting, these specimens will readily pass through a 45/50 ground-glass joint of a distillation kettle. The total surface area of a circular specimen is given by the following equation:

$$A = \frac{3.14 (D^2 - d^2)}{2} + (t)(3.14)(D) + (t)(3.14)(d)$$

where:

t = thickness.

D = diameter of the specimen

d = diameter of the mounting hole.

If the hole is completely covered by the mounting support, the last term in the equation, (t)(3.14)(d), is omitted.

4.5.1 All coupons should be measured carefully to permit accurate calculation of the exposed areas. An area calculation accurate to $\pm 1\%$ is usually adequate.

4.5.2 More uniform results may be expected if a substantial layer of metal is removed from the coupons prior to testing the corrosivity of the waste. This can be accomplished by chemical treatment (pickling), by electrolytic removal, or by grinding with a coarse abrasive. At least 0.254 mm (0.001 in.) or 2-3 mg/cm² should be removed. Final surface treatment should include finishing with #120 abrasive paper or cloth. Final cleaning consists of scrubbing with bleach-free scouring powder, followed by rinsing in distilled water and then in acetone or methanol, and finally by air-drying. After final cleaning, the coupon should be stored in a desiccator until used.

4.5.3 The minimum ratio of volume of waste to area of the metal coupon to be used in this test is 40 mL/cm².

5.0 REAGENTS

5.1 Sodium hydroxide (NaOH), 20%: Dissolve 200 g NaOH in 800 mL Type II water and mix well.

5.2 Zinc dust.

5.3 Hydrochloric acid (HCl): Concentrated.

5.4 Stannous chloride (SnCl₂)

5.5 Antimony chloride (SbCl₃).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

This method does not provide sample collection, preservation, and handling guidelines.

7.0 PROCEDURE

7.1 Assemble the test apparatus as described in Sec. 4.0, above.

7.2 Fill the container with the appropriate amount of waste.

7.3 Begin agitation at a rate sufficient to ensure that the liquid is kept well mixed and homogeneous.

7.4 Using the heating device, bring the temperature of the waste to 55 °C (130 °F).

7.5 An accurate rate of corrosion is not required; only a determination as to whether the rate of corrosion is less than or greater than 6.35 mm per year is required. A 24-hr test period should be ample to determine whether or not the rate of corrosion is > 6.35 mm per year.

7.6 In order to determine accurately the amount of material lost to corrosion, the coupons have to be cleaned after immersion and prior to weighing. The cleaning procedure should remove all products of corrosion while removing a minimum of sound metal. Cleaning methods can be divided into three general categories: mechanical, chemical, and electrolytic

7.6.1 Mechanical cleaning includes scrubbing, scraping, brushing, and ultrasonic procedures. Scrubbing with a bristle brush and mild abrasive is the most popular of these methods. The others are used in cases of heavy corrosion as a first step in removing heavily encrusted corrosion products prior to scrubbing. Care should be taken to avoid removing sound metal.

7.6.2 Chemical cleaning implies the removal of material from the surface of the coupon by dissolution in an appropriate solvent. Solvents such as acetone, dichloromethane, and alcohol are used to remove oil, grease, or resinous materials and are used prior to immersion to remove the products of corrosion. Solutions suitable for removing corrosion from the steel coupon are:

<u>Solution</u>	<u>Soaking Time</u>	<u>Temperature</u>
20% NaOH + 200 g/L zinc dust	5 min	Boiling
Conc. HCl + 50 g/L SnCl ₂ + 20 g/L SbCl ₃	Until clean	Cold

7.6.3 Electrolytic cleaning should be preceded by scrubbing to remove loosely adhering corrosion products. One method of electrolytic cleaning that can be employed uses.

Solution:	50 g/L H ₂ SO ₄
Anode:	Carbon or lead
Cathode:	Steel coupon
Cathode current density:	20 amp/cm ² (129 amp/in ²)
Inhibitor:	2 cc organic inhibitor/liter
Temperature:	74 °C (165 °F)
Exposure Period	3 min

NOTE: Precautions must be taken to ensure good electrical contact with the coupon to avoid contamination of the cleaning solution with easily reducible metal ions and to ensure that inhibitor decomposition has not occurred. Instead of a proprietary inhibitor, 0.5 g/L of either diorthotolyl thiourea or quinolin ethiodide can be used.

7.7 Whatever treatment is employed to clean the coupons, its effect in removing sound metal should be determined by using a blank (i.e., a coupon that has not been exposed to the waste). The blank should be cleaned along with the test coupon and its waste loss subtracted from that calculated for the test coupons.

7.8 After corroded specimens have been cleaned and dried, they are reweighed. The weight loss is employed as the principal measure of corrosion. Use of weight loss as a measure of corrosion requires making the assumption that all weight loss has been due to generalized corrosion and not localized pitting. In order to determine the corrosion rate for the purpose of this regulation, the following formula is used:

Corrosion Rate (mmpy) = (weight loss x 87 60)/(area x time x metal density) (Reference 1),
then,

$$\text{Corrosion Rate (mmpy)} = \frac{\text{weight loss} \times 11\,145}{\text{area} \times \text{time}}$$

where: weight loss is in milligrams,
area is in square centimeters,
time is in hours,
metal density of SAE type 1020 steel = 7.86 g/cm³, and
corrosion rate is in millimeters per year (mmpy)

8.0 QUALITY CONTROL

8.1 All quality control data should be filed and available for auditing.

8.2 Duplicate samples should be analyzed on a routine basis.

9.0 METHOD PERFORMANCE

9.1 No data provided.

10.0 REFERENCES

1. National Association of Corrosion Engineers, "Laboratory Corrosion Testing of Metals for the Process Industries," NACE Standard TM 01 69 (1972 Revision), NACE, 3400 West Loop South, Houston, TX 77027.

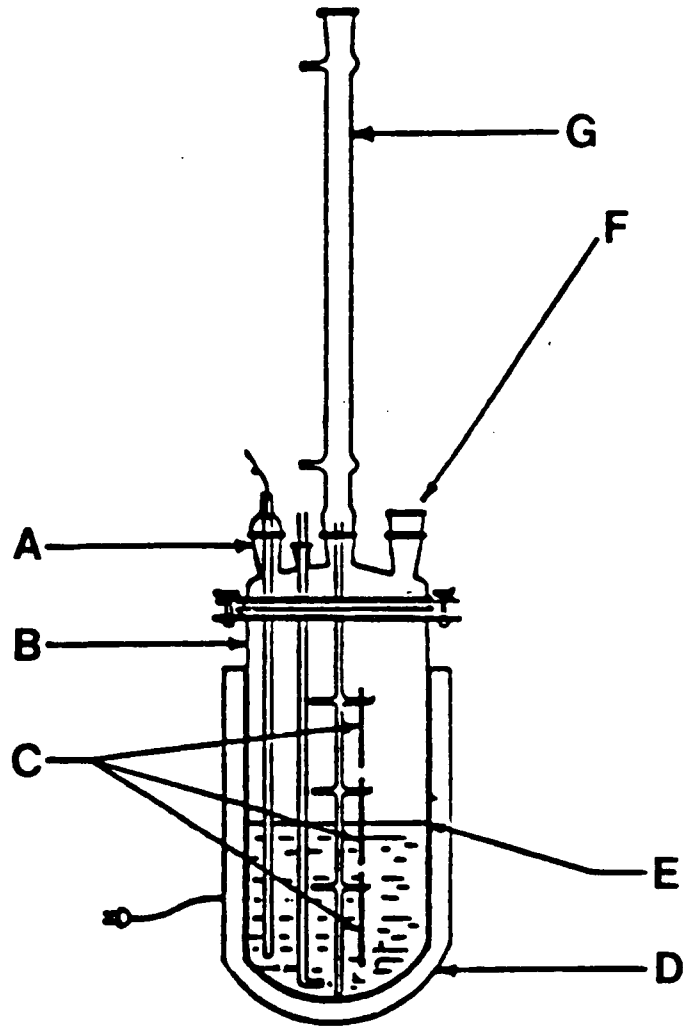
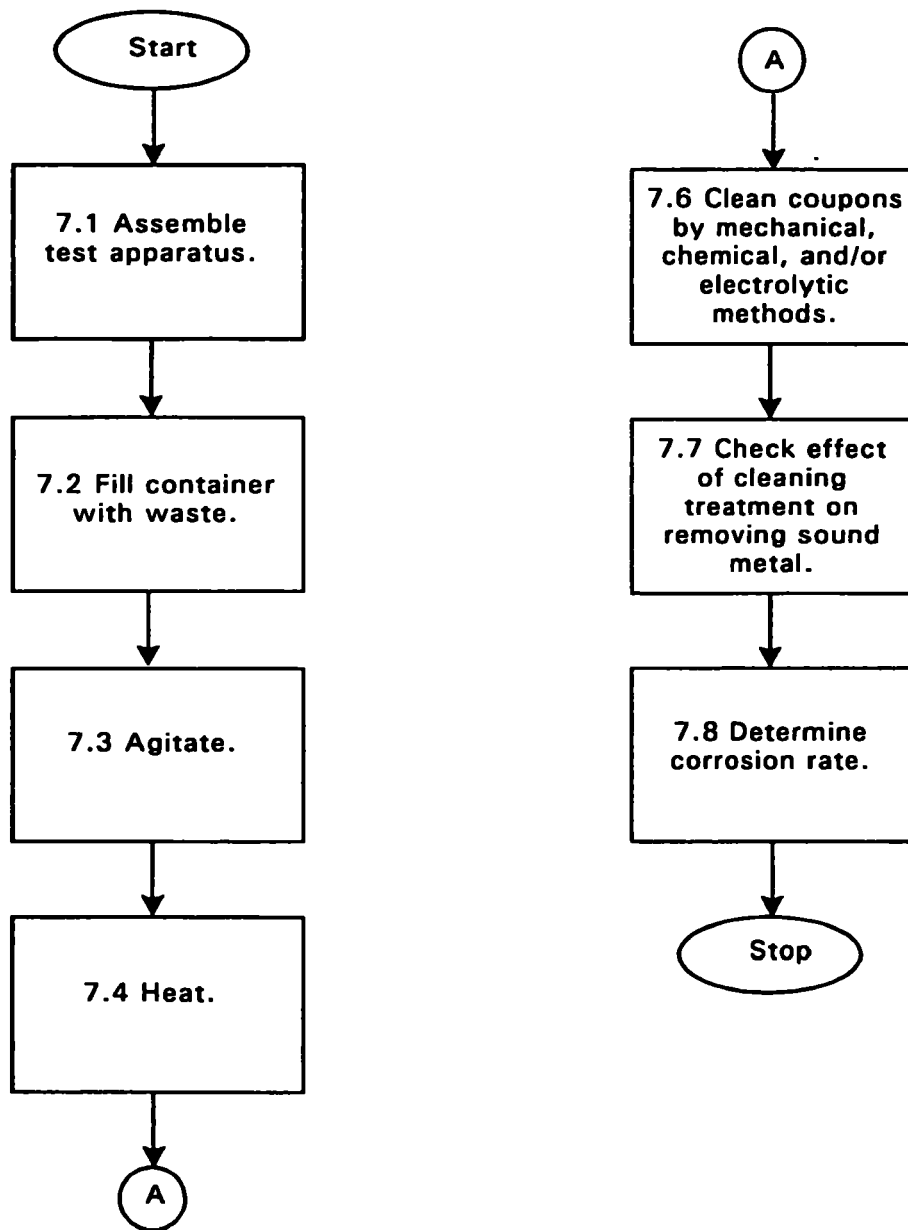


Figure 1. Typical resin flask that can be used as a versatile and convenient apparatus to conduct simple immersion tests. Configuration of the flask top is such that more sophisticated apparatus can be added as required by the specific test being conducted. A = thermowell, B = resin flask, C = specimens hung on supporting device, D = heating mantle, E = liquid interface, F = opening in flask for additional apparatus that may be required, and G = reflux condenser.

**METHOD 1110
CORROSIVITY TOWARD STEEL**



8.3 Toxicity

This chapter addresses procedures for required method-defined parameters, where the analytical result is wholly dependant on the process used to make the measurement. Examples include the use of the toxicity characteristic leaching procedure (TCLP) to prepare a leachate, and the flash point, pH, paint filter liquids, and corrosivity tests. In these instances, changes to the specific methods may change the end result and incorrectly identify a waste as nonhazardous. Therefore, when the measurement of such method-defined parameters is required by regulation, those methods are not subject to the flexibility afforded in other SW-846 methods (such as described in the Disclaimer and Chapter Two of this manual).

The following methods are found in Sec. 8.3 of this chapter:

Method 1310B:	Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test
Method 1311:	Toxicity Characteristic Leaching Procedure

METHOD 1310B

EXTRACTION PROCEDURE (EP) TOXICITY TEST METHOD AND STRUCTURAL INTEGRITY TEST

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine whether a waste exhibits the characteristic of Extraction Procedure Toxicity.

1.2 The procedure may also be used to simulate the leaching which a waste may undergo if disposed of in a sanitary landfill. Method 1310 is applicable to liquid, solid, and multiphase samples.

2.0 SUMMARY OF METHOD

2.1 If a representative sample of the waste contains > 0.5% solids, the solid phase of the sample is ground to pass a 9.5 mm sieve and extracted with deionized water which is maintained at a pH of 5 ± 0.2 , with acetic acid. Wastes that contain < 0.5% filterable solids are, after filtering, considered to be the EP extract for this method. Monolithic wastes which can be formed into a cylinder 3.3 cm (dia) x 7.1 cm, or from which such a cylinder can be formed which is representative of the waste, may be evaluated using the structural integrity procedure instead of being ground to pass a 9.5-mm sieve.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Extractor - For purposes of this test, an acceptable extractor is one that will impart sufficient agitation to the mixture to (1) prevent stratification of the sample and extraction fluid and (2) ensure that all sample surfaces are continuously brought into contact with well-mixed extraction fluid. Examples of suitable extractors are shown in Figures 1 through 3 of this method and are available from: Associated Designs & Manufacturing Co., Alexandria, Virginia; Glas-Col Apparatus Co., Terre Haute, Indiana; Millipore, Bedford, Massachusetts; and Rexnord, Milwaukee, Wisconsin.

4.2 pH meter or pH controller - Accurate to 0.05 pH units with temperature compensation.

4.3 Filter holder - Capable of supporting a 0.45- μ m filter membrane and of withstanding the pressure needed to accomplish separation. Suitable filter holders range from simple vacuum units to relatively complex systems that can exert up to 5.3 kg/cm² (75 psi) of pressure. The type

of filter holder used depends upon the properties of the mixture to be filtered. Filter holders known to EPA and deemed suitable for use are listed in Table 1.

4.4 Filter membrane - Filter membrane suitable for conducting the required filtration shall be fabricated from a material that (1) is not physically changed by the waste material to be filtered and (2) does not absorb or leach the chemical species for which a waste's EP extract will be analyzed. Table 2 lists filter media known to the Agency to be suitable for solid waste testing.

4.4.1 In cases of doubt about physical effects on the filter, contact the filter manufacturer to determine if the membrane or the prefilter is adversely affected by the particular waste. If no information is available, submerge the filter in the waste's liquid phase. A filter that undergoes visible physical change after 48 hours (i.e., curls, dissolves, shrinks, or swells) is unsuitable for use.

4.4.2 To test for absorption or leaching by the filter:

4.4.2.1 Prepare a standard solution of the chemical species of interest.

4.4.2.2 Analyze the standard for its concentration of the chemical species.

4.4.2.3 Filter the standard and reanalyze. If the concentration of the filtrate differs from that of the original standard, then the filter membrane leaches or absorbs one or more of the chemical species and is not usable in this test method

4.5 Structural integrity tester - A device meeting the specifications shown in Figure 4 and having a 3.18-cm (1.25-in) diameter hammer weighing 0.33 kg (0.73 lb) with a free fall of 15.24 cm (6 in) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA 22314, as Part No. 125, or it may be fabricated to meet these specifications.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Acetic acid (0.5N), CH_3COOH . This can be made by diluting concentrated glacial acetic acid (17.5N) by adding 57 mL glacial acetic acid to 1,000 mL of water and diluting to 2 liters. The glacial acetic acid must be of high purity and monitored for impurities.

5.4 Analytical standards should be prepared according to the applicable analytical methods.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Preservatives must not be added to samples

6.2 Samples can be refrigerated if it is determined that refrigeration will not affect the integrity of the sample.

7.0 PROCEDURE

7.1 If the waste does not contain any free liquid, go to Step 7.9. If the sample is liquid or multiphase, continue as follows. Weigh filter membrane and prefilter to ± 0.01 g. Handle membrane and prefilters with blunt curved-tip forceps or vacuum tweezers, or by applying suction with a pipet

7.2 Assemble filter holder, membranes, and prefilters following the manufacturer's instructions. Place the 0.45- μ m membrane on the support screen and add prefilters in ascending order of pore size. Do not prewet filter membrane.

7.3 Weigh out a representative subsample of the waste (100 g minimum).

7.4 Allow slurries to stand, to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.5 Wet the filter with a small portion of the liquid phase from the waste or from the extraction mixture. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops. This liquid will constitute part or all of the extract (refer to Step 7.16). The liquid should be refrigerated until time of analysis.

NOTE: Oil samples or samples containing oil are treated in exactly the same way as any other sample. The liquid portion of the sample is filtered and treated as part of the EP extract. If the liquid portion of the sample will not pass through the filter (usually the case with heavy oils or greases), it should be carried through the EP extraction as a solid.

7.6 Remove the solid phase and filter media and, while not allowing them to dry, weigh to ± 0.01 g. The wet weight of the residue is determined by calculating the weight difference between the weight of the filters (Step 7.1) and the weight of the solid phase and the filter media.

7.7 The waste will be handled differently from this point on, depending on whether it contains more or less than 0.5% solids. If the sample appears to have $< 0.5\%$ solids, determine the percent solids exactly (see Note below) by the following procedure:

7.7.1 Dry the filter and residue at 80 °C until two successive weighings yield the same value.

7.7.2 Calculate the percent solids, using the following equation:

$$\frac{\text{weight of filtered solid and filters} - \text{tared weight of filters}}{\text{initial weight of waste material}} \times 100 = \% \text{ solids}$$

NOTE: This procedure is used only to determine whether the solid must be extracted or whether it can be discarded unextracted. It is not used in calculating the amount of water or acid to use in the extraction step. Do not extract solid material that has been dried at 80 °C. A new sample will have to be used for extraction if a percent solids determination is performed.

7.8 If the solid constitutes < 0.5% of the waste, discard the solid and proceed immediately to Step 7.17, treating the liquid phase as the extract.

7.9 The solid material obtained from Step 7.5 and all materials that do not contain free liquids shall be evaluated for particle size. If the solid material has a surface area per g of material $\geq 3.1 \text{ cm}^2$ or passes through a 9.5-mm (0.375-in.) standard sieve, the operator shall proceed to Step 7.11. If the surface area is smaller or the particle size larger than specified above, the solid material shall be prepared for extraction by crushing, cutting, or grinding the material so that it passes through a 9.5-mm (0.375-in.) sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described in Step 7.10

7.10 Structural integrity procedure (SIP)

7.10.1 Cut a 3.3-cm diameter by 7.1-cm long cylinder from the waste material. If the waste has been treated using a fixation process, the waste may be cast in the form of a cylinder and allowed to cure for 30 days prior to testing.

7.10.2 Place waste into sample holder and assemble the tester. Raise the hammer to its maximum height and drop. Repeat 14 additional times.

7.10.3 Remove solid material from tester and scrape off any particles adhering to sample holder. Weigh the waste to the nearest 0.01 g and transfer it to the extractor.

7.11 If the sample contains > 0.5% solids, use the wet weight of the solid phase (obtained in Step 7.6) to calculate the amount of liquid and acid to employ for extraction by using the following equation:

$$W = W_t - W_f$$

where :

W = Wet weight in g of solid to be charged to extractor.

W_t = Wet weight in g of filtered solids and filter media.

W_f = Weight in g of tared filters.

If the waste does not contain any free liquids, 100 g of the material will be subjected to the extraction procedure.

7.12 Place the appropriate amount of material (refer to Step 7.11) into the extractor and add 16 times its weight with water.

7.13 After the solid material and water are placed in the extractor, the operator shall begin agitation and measure the pH of the solution in the extractor. If the pH is > 5.0 , the pH of the solution should be decreased to 5.0 ± 0.2 by slowly adding 0.5N acetic acid. If the pH is ≤ 5.0 , no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction, and, if the pH rises above 5.2, 0.5N acetic acid should be added to bring the pH down to 5.0 ± 0.2 . However, in no event shall the aggregate amount of acid added to the solution exceed 4 mL of acid per g of solid. The mixture should be agitated for 24 hours and maintained at 20-40 °C (68-104 °F) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller, manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123, or its equivalent, in conjunction with a metering pump and reservoir of 0.5N acetic acid. If such a system is not available, the following manual procedure shall be employed.

NOTE: Do not add acetic acid too quickly. Lowering the pH to below the target concentration of 5.0 could affect the metal concentrations in the leachate.

7.13.1 A pH meter should be calibrated in accordance with the manufacturer's specifications.

7.13.2 The pH of the solution should be checked, and, if necessary, 0.5 N acetic acid should be manually added to the extractor until the pH reaches 5.0 ± 0.2 . The pH of the solution should be adjusted at 15-, 30-, and 60-minute intervals, moving to the next longer interval if the pH does not have to be adjusted > 0.5 pH units.

7.13.3 The adjustment procedure should be continued for at least 6 hours.

7.13.4 If, at the end of the 24-hour extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 mL per g of solids) has not been added, the pH should be adjusted to 5.0 ± 0.2 and the extraction continued for an additional 4 hours, during which the pH should be adjusted at 1-hour intervals.

7.14 At the end of the extraction period, water should be added to the extractor in an amount determined by the following equation:

$$V = (20)(W) - 16(W) - A$$

where:

V = mL water to be added.

W = Weight in g of solid charged to extractor.

A = mL of 0.5N acetic acid added during extraction.

7.15 The material in the extractor should be separated into its component liquid and solid phases in the following manner:

7.15.1 Allow slurries to stand to permit the solid phase to settle (wastes that are slow to settle may be centrifuged prior to filtration) and set up the filter apparatus (refer to Steps 4.3 and 4.4).

7.15.2 Wet the filter with a small portion of the liquid phase from the waste or from the extraction mixture. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops.

7.16 The liquids resulting from Steps 7.5 and 7.15 should be combined. This combined liquid (or waste itself, if it has < 0.5% solids, as noted in Step 7.8) is the extract.

7.17 The extract is then prepared and analyzed using the appropriate analytical methods described in Chapters Three and Four of this manual.

NOTE: If the EP extract includes two phases, concentration of contaminants is determined by using a simple weighted average. For example: An EP extract contains 50 mL of oil and 1,000 mL of an aqueous phase. Contaminant concentrations are determined for each phase. The final contamination concentration is taken to be:

$$\frac{(50 \times \text{contaminant conc. in oil}) + (1,000 \times \text{contaminant conc. of aqueous phase})}{1050}$$

NOTE: In cases where a contaminant was not detected, use the MDL in the calculation. For example, if the MDL in the oily phase is 100 mg/L and 1 mg/L in the aqueous phase, the reporting limit would be 6 mg/L (rounded to the nearest mg). If the regulatory threshold is 5 mg/L, the waste may be EP toxic and results of the analysis are inconclusive.

8.0 QUALITY CONTROL

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

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

8.3 All quality control measures described in Chapter One and in the referenced analytical methods should be followed.

9.0 METHOD PERFORMANCE

9.1 The data tabulated in Table 3 were obtained from records of state and contractor laboratories and are intended to show the precision of the entire method (1310 plus analysis method).

10.0 REFERENCES

- 1.** Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.
- 2.** 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM. Philadelphia, PA, 1985; D1193-77.
- 3.** Gaskill, A , Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

TABLE 1. EPA-APPROVED FILTER HOLDERS

Manufacturer	Size	Model No.	Comments
<u>Vacuum Filters</u>			
Gelman	47 mm	4011	
Nalgene	500 mL	44-0045	Disposable plastic unit, including prefilter, filter pads, and reservoir; can be used when solution is to be analyzed for inorganic constituents.
Nuclepore	47 mm	410400	
Millipore	47 mm	XX10 047 00	
<u>Pressure Filters</u>			
Nuclepore	142 mm	425900	
Micro Filtration Systems	142 mm	302300	
Millipore	142 mm	YT30 142 HW	

TABLE 2. EPA-APPROVED FILTRATION MEDIA

Supplier	Filter to be used for aqueous systems	Filter to be used for organic systems
<u>Coarse prefilter</u>		
Gelman	61631, 61635	61631, 61635
Nuclepore	210907, 211707	210907, 211707
Millipore	AP25 035 00, AP25 127 50	AP25 035 00, AP25 127 50
<u>Medium prefilters</u>		
Gelman	61654, 61655	
Nuclepore	210905, 211705	210905, 211705
Millipore	AP20 035 00, AP20 124 50	AP20 035 00, AP20 124 50
<u>Fine prefilters</u>		
Gelman	64798, 64803	64798, 64803
Nuclepore	210903, 211703	210903, 211703
Millipore	AP15 035 00, AP15 124 50	AP15 035 00, AP15 124 50
<u>Fine filters (0.45 µm)</u>		
Gelman	63069, 66536	60540 or 66149, 66151
Pall	NX04750, NX14225	
Nuclepore	142218	142218 ^a
Millipore	HAWP 047 00, HAWP 142 50	FHUP 047 00, FHLP 142 50
Selas	83485-02, 83486-02	83485-02, 83486-02

^a Susceptible to decomposition by certain polar organic solvents.

**TABLE 3. PRECISIONS OF EXTRACTION-ANALYSIS
PROCEDURES FOR SEVERAL ELEMENTS**

Element	Sample Matrix	Analysis Method	Laboratory Replicates
Arsenic	1. Auto Fluff	7060	1.8, 1.5 µg/L
	2. Barrel sludge	7060	0.9, 2.6 µg/L
	3. Lumber treatment company sediment	7060	28, 42 mg/L
Barium	1. Lead smelting emission control dust	6010	0.12, 0.12 mg/L
	2. Auto Fluff	7081	791, 780 µg/L
	3. Barrel Sludge	7081	422, 380 µg/L
Cadmium	1. Lead smelting emission control dust	3010/7130	120, 120 mg/L
	2. Wastewater treatment sludge from electroplating	3010/7130	360, 290 mg/L
	3. Auto fluff		
	4. Barrel sludge	7131	470, 610 µg/L
	5. Oil refinery tertiary pond sludge	7131	1100, 890 µg/L
Chromium		7131	3.2, 1.9 µg/L
	1. Wastewater treatment sludge from electroplating	3010/7190	1.1, 1.2 mg/L
	2. Paint primer		
	3. Paint primer filter	7191	61, 43 µg/L
	4. Lumber treatment company sediment	7191	–
Mercury		7191	0.81, 0.89 mg/L
	5. Oil refinery tertiary pond sludge	7191	–
	1. Barrel sludge	7470	0.15, 0.09 µg/L
	2. Wastewater treatment sludge from electroplating	7470	1.4, 0.4 µg/L
	3. Lead smelting emission control dust	7470	0.4, 0.4 µg/L

TABLE 3 (Continued)

Element	Sample Matrix	Analysis Method	Laboratory Replicates
Lead	1. Lead smelting emission control dust	3010/7420	940, 920 mg/L
	2. Auto fluff	7421	1540, 1490 µg/L
	3. Incinerator ash	7421	1000, 974 µg/L
	4. Barrel sludge	7421	2550, 2800 µg/L
	5. Oil refinery tertiary pond sludge	7421	31, 29 µg/L
Nickel	1. Sludge	7521	2260, 1720 µg/L
	2. Wastewater treatment sludge from electroplating	3010/7520	130, 140 mg/L
Chromium (VI)	1. Wastewater treatment sludge from electroplating	7196	18, 19 µg/L

FIGURE 1.
EXTRACTOR

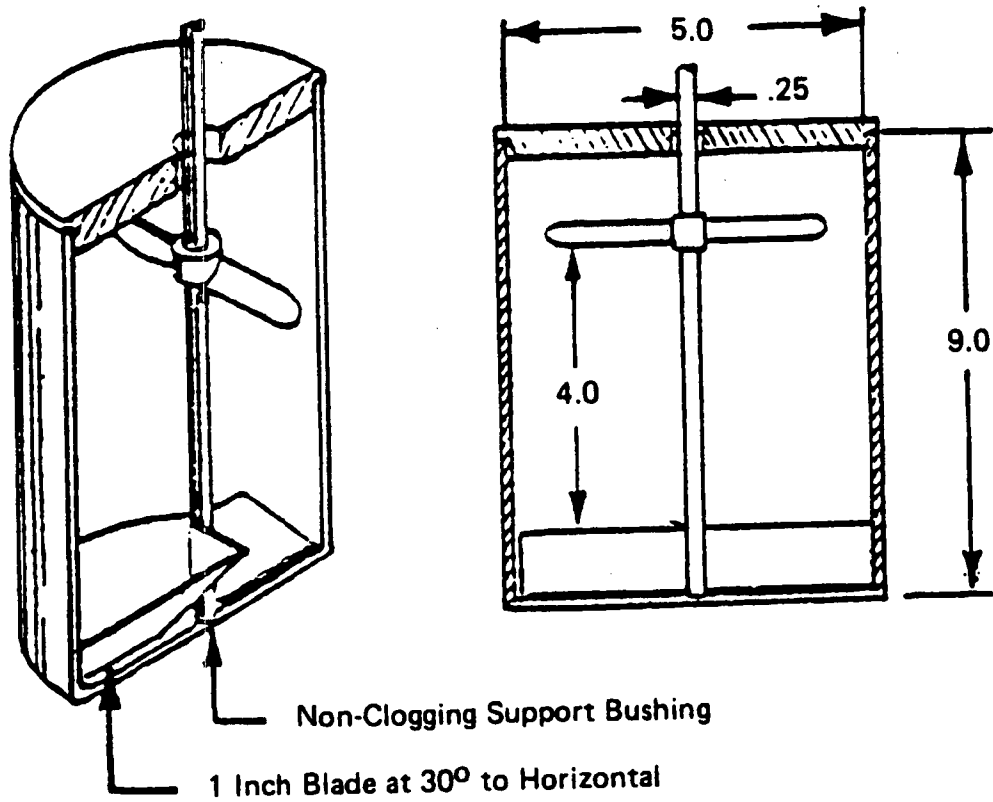


FIGURE 2.
ROTARY EXTRACTOR

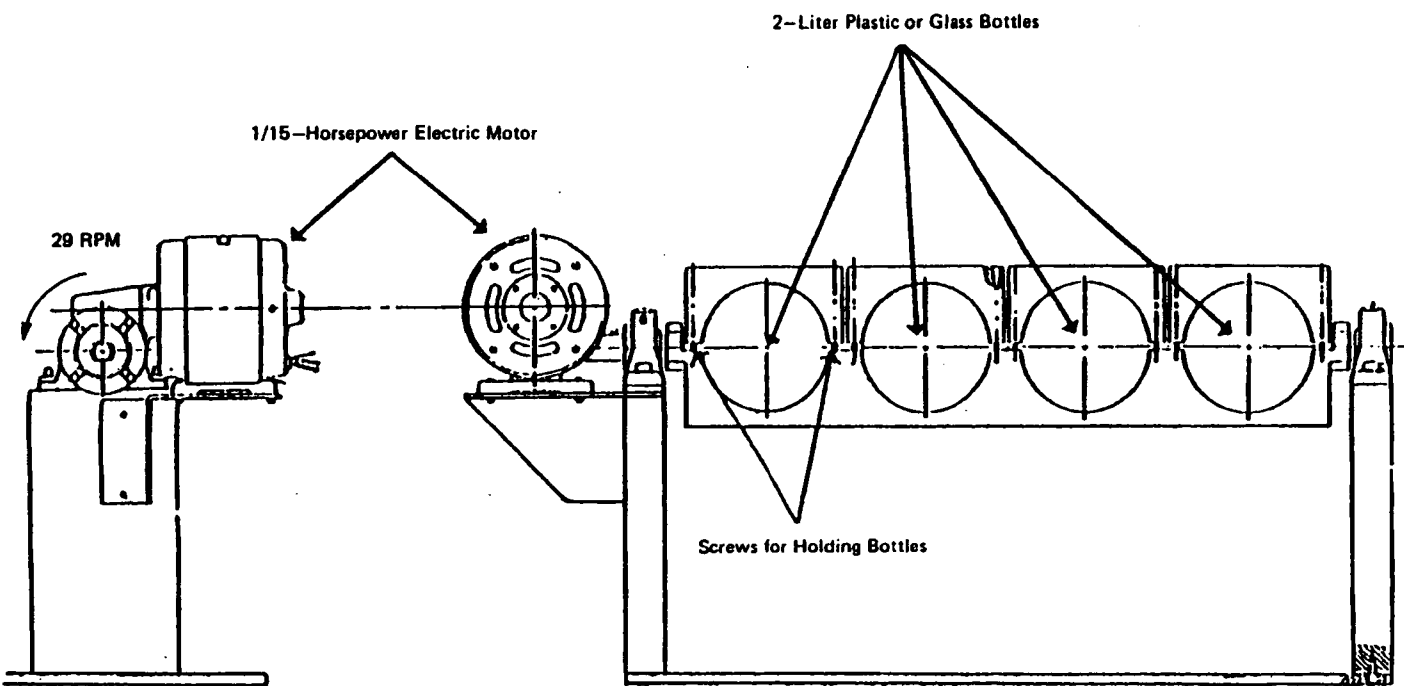


FIGURE 3.
EPRI EXTRACTOR

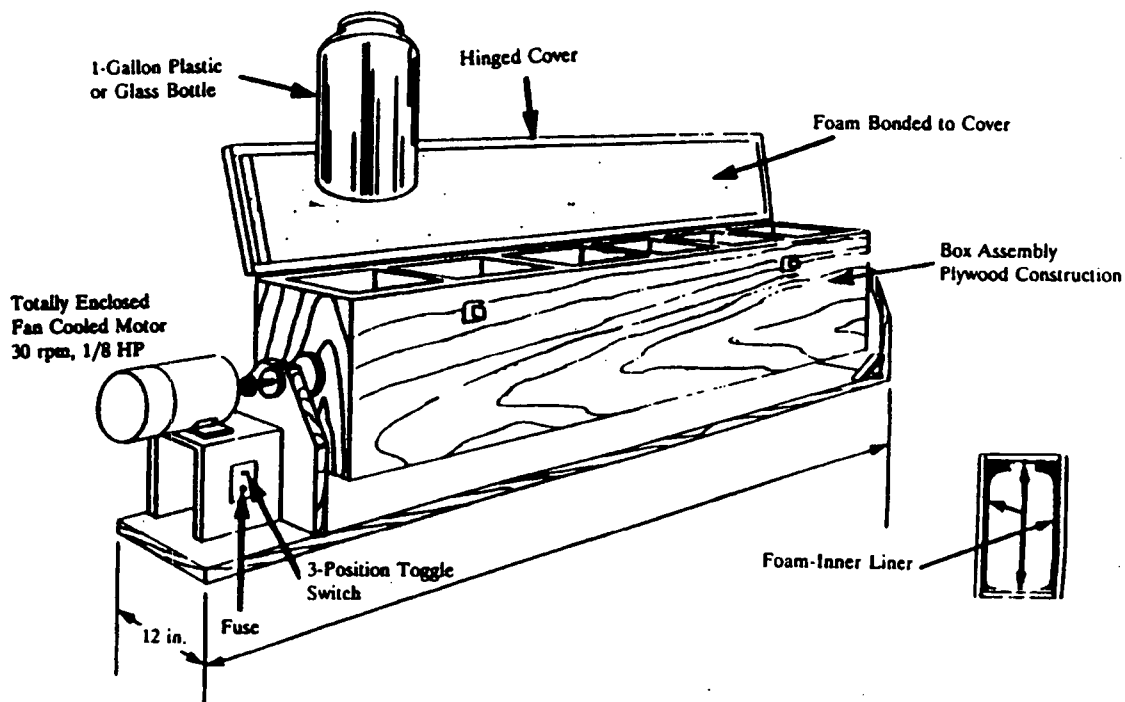
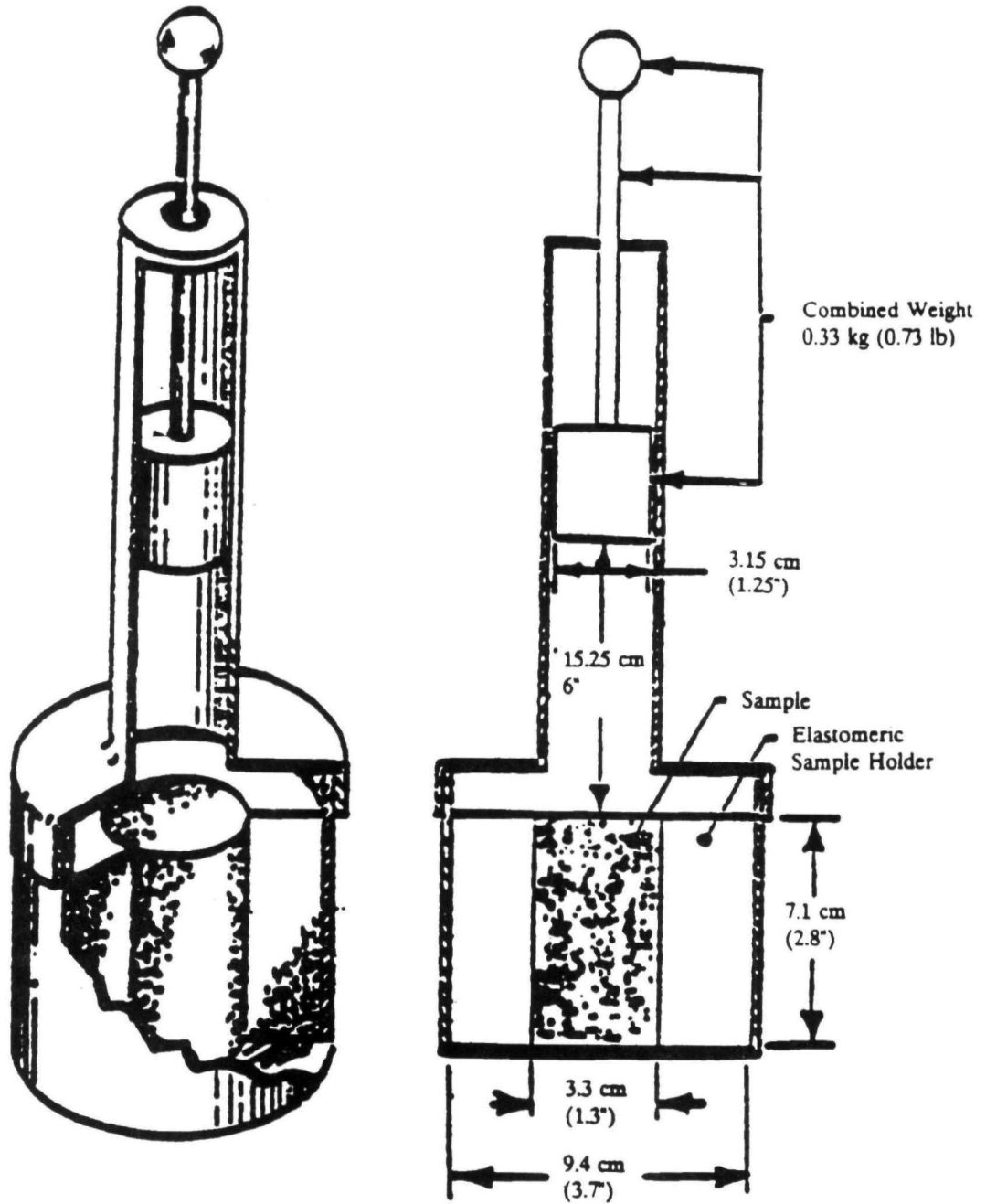
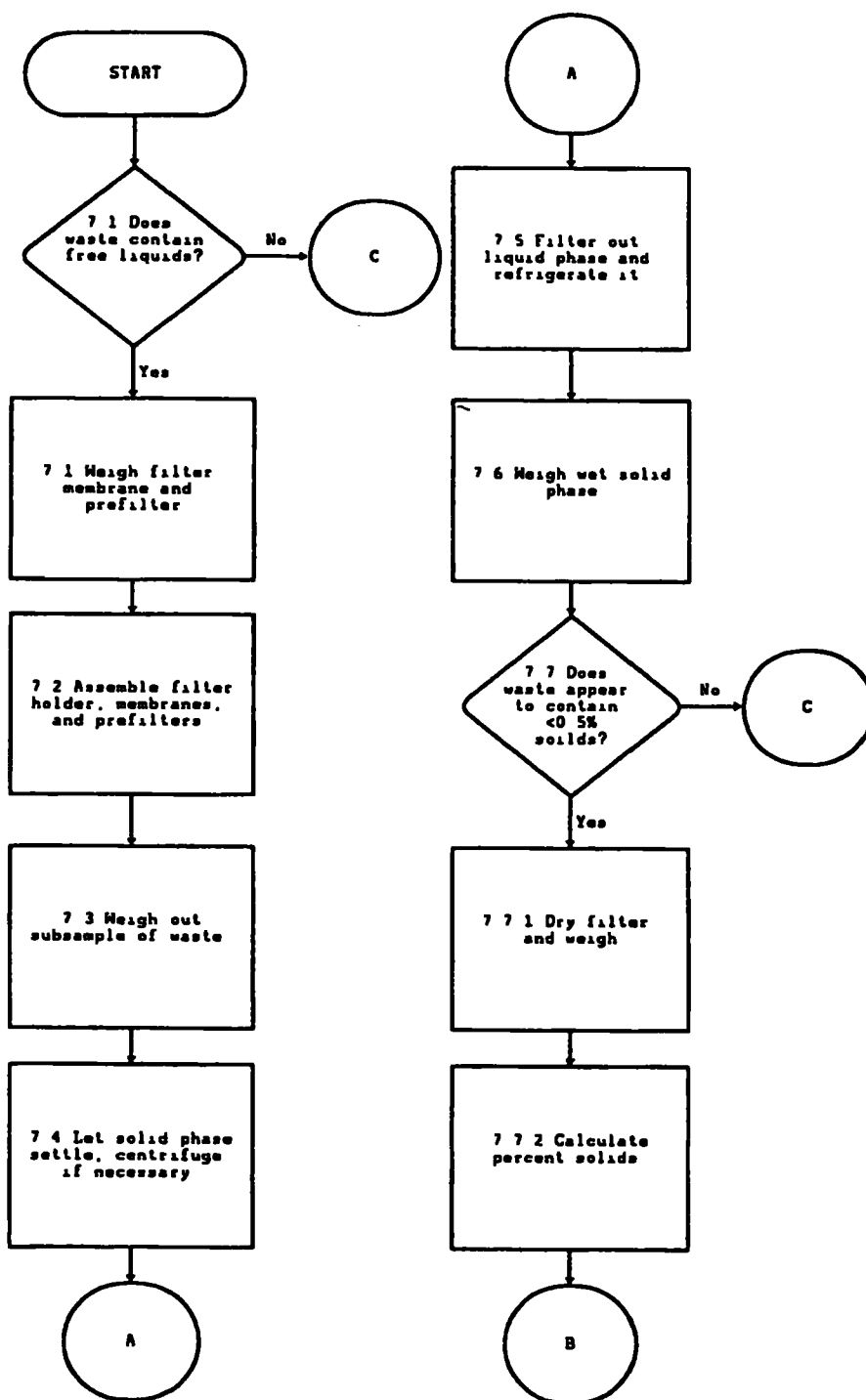


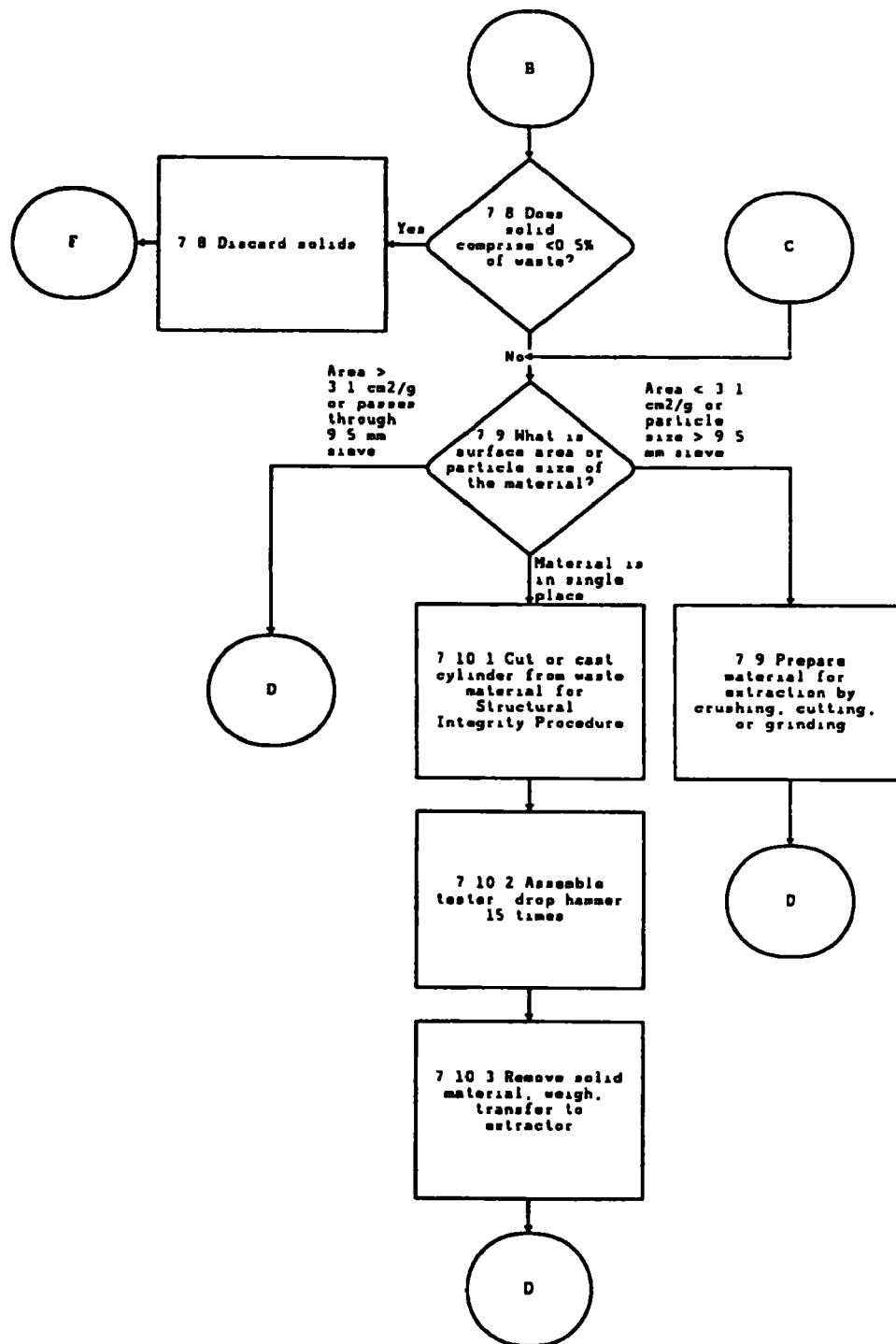
FIGURE 4.
COMPACTION TESTER



METHOD 1310B
EXTRACTION PROCEDURE (EP) TOXICITY TEST METHOD
AND STRUCTURAL INTEGRITY TEST



METHOD 1310B
(Continued)



METHOD 1310B
(Continued)

